New Jersey
Sludge Sampling and
Analytical Guidance Document

Prepared by: New Jersey Department of Environmental Protection
Division of Water Quality
Bureau of Ground Water, Residuals and Permit Administration
May 2021

CONTENTS

Chapter 7: Data Handling Procedures

Appendices

FORWARD / ACKNOWLEDGEMENTS

The New Jersey Department of Environmental Protection has compiled this Guidance Document for treatment plant operators, analysts, data users or other interested parties to assist in the collection of reliable and consistent sludge samples. The information contained in this Guidance Document may be employed for the evaluation of sludge as specified in regulations issued under the New Jersey Water Pollution Control Act.

This Guidance Document has been prepared, in part, pursuant to N.J.S.A. 13:1D-111 to 1D-113, to assist with obtaining accurate sludge analytical results when information on sludge quality is required. For example, all permit applications submitted under the New Jersey Pollutant Discharge Elimination System (NJPDES) require submission of a sludge sampling plan when sludge is anticipated to be generated or processed. In addition, all NJPDES permits require establishment of a sludge sampling location that is representative of the sludge generated for use or disposal. Thus, this Guidance Document is intended to be used by applicants to facilitate preparation of permit applications as well as monitoring reports, and the review thereof by the Department.

Because this Guidance Document by necessity condenses and summarizes statutes, regulations, and other documents, it may not always precisely reflect all the requirements set forth in same. In the case of any inconsistency between this Document and any statutes, regulations, or policy determinations based upon same, the requirements of the statutes, regulations, or policy determinations shall prevail. Accordingly, this Guidance Document should not be used as a substitute for a thorough analysis of the law and the facts as they apply to any specific project or proposal.

The Department does not intend to restrict the use of new analytical techniques. Advances in technologies applicable to the sampling and analysis of environmental media outpace the ability of the Department to promulgate revisions to rules or this Guidance Document. The Department emphasizes that the ultimate responsibility for producing reliable analytical results lies with the entity subject to State regulation. Thus, members of the regulated community are advised to consult with knowledgeable laboratory personnel when choosing the most appropriate suite of analytical methods.

In summary, the procedures and methods herein provide guidance to the analyst and regulated community in making judgments necessary to submit permit applications and to generate data that meet the data quality objectives for the intended use of the results.

The Department would like to thank the Environmental Laboratory Advisory Committee and the New Jersey Department of Environmental Protection's Office of Quality Assurance for assistance in development of this Guidance Document. Questions or comments on this Guidance Document may be directed to the New Jersey Department of Environmental Protection, Division of Water Quality, Bureau of Ground Water, Residuals and Permit Administration at (609) 984 - 4428 or by email at DWQ_Residuals@dep.nj.gov.

CHAPTER 1
OVERVIEW

Introduction

The New Jersey Department of Environmental Protection (Department) administers a regulatory program for the use and management of residuals generated by domestic and industrial treatment works. Of fundamental importance is the need to control sludge quality. Under applicable laws, the Department is directed to issue permits to limit concentrations of heavy metals, pesticides, organic chemicals and other contaminants in sludge in conformance with land-based sludge management criteria.

The Sludge Quality Assurance Regulations (SQAR), N.J.A.C. 7:14C, were initially promulgated in October 1979. With the SQAR, the Department embarked on a major program of monitoring the quality and quantity of sludge generated throughout the State by domestic and industrial treatment works. For clarification, SQAR also applies to out-of-state sludge generators if they transport sludge into the State of New Jersey for use or disposal. The SQAR have been in effect for more than 40 years, and the information submitted has been extremely useful to the Department in evaluating sludge management plans, and to the generators in developing appropriate sludge management alternatives.

On February 19, 1993, under the authority of Section 405(d) and (e) of the Clean Water Act, the United States Environmental Protection Agency (USEPA) promulgated Federal sludge management regulations at 40 CFR Part 503. These Federal regulations establish general requirements, pollutant limits, management practices, and operational standards for the final use or disposal of sewage sludge generated during the treatment of domestic sewage in a treatment works. The USEPA established a monitoring frequency from annual to monthly based upon the metric tons of sewage sludge generated by the domestic treatment works on an annual basis. The Department subsequently incorporated the provisions of the Federal rule for land application into State regulation under the New Jersey Pollutant Discharge Elimination System (NJPDES) at N.J.A.C. 7:14A-20. The NJPDES rule incorporates the standards, management practices, monitoring, reporting, and recordkeeping requirements specific to certain sludge use or disposal alternatives. SQAR, on the other hand, addresses the monitoring and reporting requirements for all sludge generators as well as sludge sampling and analytical requirements.

It is the purpose of this Guidance Document to provide a road map through the many State and Federal statutes and regulations that include requirements for sludge sampling and analysis. In addition, this Guidance Document may be used to provide assistance on the sampling and analysis of sludge as may be required under SQAR or under individual or general permits issued for sludge management under NJPDES. Sampling and analysis is an integral part of monitoring the quality of sludge going to ultimate management (for example, land application). Sludge samples must be acquired in a manner that will not compromise subsequent analysis, and the final validated procedures must be reliable and consistently performed. Sampling must ensure that the material collected is representative of the sludge being removed for use or disposal.

The sample container, transport conditions, holding times and handling procedures must be controlled to ensure that there is no gain or loss of analyte prior to receipt by the certified laboratory. Similarly, at the laboratory, the samples must not be contaminated or lost, and the analyte concentrations must be determined using techniques of adequate selectivity and sensitivity to ensure reliable and useful results.

To accomplish the above goals, the Department convened a working group of sludge and laboratory professionals to gather information on the best ways to achieve reliable sludge monitoring results. Development of this Guidance Document is intended to ensure that the regulated community has, in one place, the information necessary to facilitate the preparation and analysis of sludge samples to maximize the integrity and reliability of data submissions.

CHAPTER 2
REPORTING REQUIREMENTS

Domestic treatment works

Sludge quality and characteristics vary considerably from one plant to another and even from time to time within a given plant. Chemicals may be added as conditioning agents, thereby increasing the quantity of solids. Without pretreatment for removal of pollutants, industrial users of publicly owned treatment works may be a major source of such pollutants. Industrial users are not the only source, however, and it should not be presumed that all pollutants will be removed by a pretreatment program for industrial users. Domestic wastes typically have quantities of pollutants from the use of chemical products in the home, from exposure to metallic plumbing systems, and from street runoff in areas of combined sewers.

Sludge characteristics may vary for many other reasons. Wastewater characteristics may change seasonally (because of industrial or recreational uses), with corresponding changes in the sludge quality. Process upsets, equipment malfunctions, or industrial spills also may change sludge properties. In order to characterize sludge, therefore, it is necessary to test periodically rather than rely on one test result.

All domestic treatment works are required to analyze for the 18 parameters found in Appendix, Table I, of the SQAR and as replicated in Appendix A, "Analytical Parameters, Methods, Holding Times, and Target Reporting Level", of this Guidance Document. These parameters include pollutants for which limits are included in 40 CFR Part 503 for land application (arsenic, cadmium, copper, lead, mercury, molybdenum, nickel, selenium, and zinc), and additional parameters which are only limited for incineration (chromium and beryllium). The Department also requires generators to submit data for nutrients (total kjeldahl nitrogen, ammonia-nitrogen, nitrate-nitrogen, total phosphorus, potassium and calcium). The metals and nutrients discussed above constitute 17 of the parameters required to be reported on a routine basis. In addition, the percent total solids of the sludge that is used or disposed must also be reported. Percent solids is required to ensure that sludge data can be converted to dry weight values. Additional contaminants, such as radionuclides, could be required on a case-by-case basis.

The frequencies with which SQAR discharge monitoring reports are required to be submitted for the above parameters are based on the size of the domestic treatment works as follows:

- Category **1**: Domestic treatment works with a permitted flow 0.099 million gallons per day (MGD) or less are required to report the above parameters one time per year.
- Category **2**: Domestic treatment works with a permitted flow from 0.1 to 0.999 MGD are required to report the above parameters two times per year.
- Category **3**: Domestic treatment works with a permitted flow from 1.0 to 4.999 MGD are required to report the above parameters four times per year.

- Category **4**: Domestic treatment works with a permitted flow equal to or greater than 5.0 MGD are required to report the above parameters twelve times per year.

Category 3 and 4 domestic treatment works are also required to perform an annual priority pollutant scan for those parameters listed in the SQAR Appendix, Tables II through VI, and as replicated in Appendix A of this Guidance Document as Tables II through VI.

Where a treatment works generates several different types of sludge (for example, liquid digested sludge and limed sludge cake), and each type of sludge is not combined for use or disposal, separate composite samples for each different type of sludge are required to be analyzed and reported. In addition, for those treatment works which take customer sludge, the Department often requires multiple monitoring locations be established to capture sludge quality for the host treatment works (for example, for pretreatment purposes) as well as for the final blend to determine compliance with land-based sludge management criteria.

It is the responsibility of the sludge generator to communicate the details of how their treatment plants are operated at the time of application for their respective NJPDES operating permits. The Department will establish monitoring locations based on the information submitted and will develop monitoring report forms to be utilized by the sludge generator. It is the responsibility of the sludge generator to notify the Department should any changes in sludge handling, or sludge use or disposal occur, which may necessitate a change in the sludge monitoring location or other permit requirements.

There are instances when reduced reporting or reduced analyses may be allowed as follows:

- A domestic treatment works which is required to obtain a NJPDES permit, but generates only domestic septage, or a small domestic treatment works that has a permitted wastewater flow of less than or equal to 20,000 gallons per day (0.020 million gallons per day) that removes their sludge to an off-site in-State treatment works treating domestic sewage, are exempt from the requirement to perform analyses. However, an annual Residuals Transfer Report is still required to track the gallons of septage or sludge removed, and the septage or sludge use or disposal site. Domestic septage is defined to mean either liquid or solid material removed from a septic tank, cesspool, portable toilet, Type III marine sanitation device, or similar treatment works that receives only domestic sewage. Domestic septage does not include liquid or solid material removed from a septic tank, cesspool, or similar treatment works that receives process wastewater and does not include grease removed from a grease trap.
- Analyses are required to be submitted on sludge removed for use or disposal. In the event that sludge is not removed from the treatment works during a monitoring period, analyses are not required to be conducted.

Industrial treatment works

SQAR serves as a useful data collection system when that data collection and reporting mechanism can be tailored to a specific industrial treatment works. For this reason the Department distinguishes between Industrial Wastewater Treatment Systems (IWTS) and Public

(drinking) Water Treatment Systems (PWTS). The reporting requirements for IWTS are more flexible than those for PWTS or domestic treatment works.

The frequency of reporting for industrial treatment works is based on the amount of sludge that is removed for use or disposal. Essentially, the larger a particular system is and the more treatment a system provides, the more frequently a generator will have to report under SQAR. The reporting categories and reporting frequencies are as follows:

- Category **10** and **6**: Any PWTS or any IWTS respectively with a sludge production greater than zero, but less than 290 dry metric tons per 365-day period. The reporting frequency for category 10 and 6 industrial treatment works is one month annually.
- Category **11** and **7**: Any PWTS or any IWTS respectively with a sludge production equal to or greater than 290, but less than 1,500 dry metric tons per 365-day period. The reporting frequency for category 11 and 7 industrial treatment works is any one calendar month in each three-month period.
- Category **12** and **8**: Any PWTS or any IWTS respectively with a sludge production equal to or greater than 1,500, but less than 15,000 dry metric tons per 365-day period. The reporting frequency for category 12 and 8 industrial treatment works is any one calendar month in each two-month period.
- Category **13** and **9**: Any PWTS or any IWTS respectively with a sludge production greater than or equal to 15,000 dry metric tons per 365-day period. The reporting frequency for category 13 and 9 industrial treatment works is every calendar month.

For IWTS, the parameters required to be analyzed and reported are based on what is "manufactured, processed, formed, repackaged, handled, used, disposed, or stored" at the facility served by the industrial treatment works. The list of parameters is found in the SQAR Appendix, Tables I through VII, and are replicated in Appendix A of this Guidance Document as Tables I through VIII. An IWTS is not required to analyze their sludge for all of the parameters in the SQAR Appendix, but is instead required to choose for analysis which parameters may be expected to be present in the sludge generated based on their knowledge of facility operations. Therefore, it is essential for IWTS operators to communicate to the Department the list of contaminates they expect may be present in the sludge generated from their operations. The Department will establish monitoring locations based on the information submitted and will develop monitoring report forms to be utilized by the sludge generator. It is the responsibility of the sludge generator to notify the Department should any changes in the manufacturing operations occur which may necessitate a change in the contaminants to be reported.

PWTS generally contain a consistent range of contaminants, although the reported levels of concentration can vary greatly. Therefore, the Department separately classifies PWTS, and limits the monitoring requirements to: the metals regulated pursuant to 40 CFR Part 503; nutrients; aluminum or iron (depending on the coagulant used); and, trihalomethanes (that is, bromoform, chloroform, dibromochloromethane and dichlorobromomethane) if the PWTS receives all or a portion of the water treated from a surface water source and chlorinates the water prior to distribution. Additional contaminants, such as radionuclides, could be required on a case-by-case basis if expected to be present.

All industrial treatment works that generate a non-hazardous sludge are required to comply with SQAR. If an IWTS does not have an individual or general NJPDES permit issued by the Department, then there may not be forms generated on which to submit the required information. Therefore, although these facilities are still required to sample, perform analyses, and maintain records, they are not required to submit reports to the Department until such time as the Department issues a SQAR general residual generator permit to the facility. However, until such time as a general residual permit is issued, these IWTS are required to maintain on file with the Department certain basic information as set forth below. In addition, these IWTS are required to resubmit the information below each time physical alterations or additions are made to the IWTS when the additions or alterations are expected to result in a change in the quality of the sludge generated or when there is a change in the sludge use or disposal practice:

1. Name, mailing address and location of the IWTS;
2. The domestic wastewater contribution as a percentage of total influent;
3. The operator's name, address, telephone number, ownership status, and status as Federal, State, private, public or other entity;
4. A description of the sludge use or disposal practices (including, where applicable, the location of any sites where sludge is transferred for treatment, use, or disposal, as well as the name of any applicator or other contractor who applies the sludge to land);
5. The annual amount of sludge generated at the industrial treatment works, the annual amount of sludge received from off-site sources and the annual amount of sludge removed for each use or disposal method for the latest 365 day period in total dry metric tons; and
6. The most recent data the IWTS may have on the quality of the sludge it generates.

The Department has developed a standard application form, Form R, for reporting this information. Based on the information submitted, the Department may designate an IWTS as a Treatment Works Treating Domestic Sewage or as a sludge-only facility and require said treatment works to submit reports under SQAR and/or to apply for a NJPDES permit. All PWTS are required to submit monitoring results to the Department and have been issued either a general or individual NJPDES permit. The Department has created standardized monitoring and reporting forms for use by all PWTS.

The question is often asked, "Is the by-product my facility generates considered a sludge subject to SQAR?" As a general rule, if chemicals are added to aid in settling in a tank, basin, pond etc., then sludge is generated and subject to SQAR. Also, as a general rule, if treatment is provided in order to meet an effluent limitation, any resultant by-product of the treatment of that wastewater is considered sludge subject to SQAR. For example, filter backwash from a drinking water authority which is hauled or discharged to the sewer without treatment is not sludge for purposes of SQAR. However, if that filter backwash is sent to a sedimentation basin for settling then the settled material is sludge subject to SQAR.

With regards to oil/water separators, generally, oil/water separators are considered a treatment unit that generates sludge. If the sludge generated in an oil/water separator is removed and

disposed as a nonhazardous waste it would be considered sludge subject to regulation under SQAR. However, if the sludge is considered to be a hazardous waste and is manifested, or if the sludge is recycled, then generally SQAR would not be applicable.

Sludge Management Operations

Any sludge management permit issued by the Department requires the permittee to limit concentrations of heavy metals, pesticides, organic chemicals and other contaminants in the sludge in conformance with the land-based sludge management criteria. At a minimum, information is required on the origin, volume and chemical characteristics of the sludge. The chemical characteristics must include, at a minimum, a dated analysis for all constituents for which limits have been established, or as may otherwise be required in a permit issued by the Department.

For land application, monitoring and reporting requirements for pollutant concentrations, pathogen density requirements, and vector attraction reduction requirements vary from quarterly to monthly based upon the amount of sludge applied to the land or prepared for application to the land as follows:

- Any person who prepares sludge and applies to the land less than 1,500 dry metric tons per 365-day period, the monitoring frequency is any one calendar month in each three-month period.
- Any person who prepares sludge and applies to the land equal to or greater than 1,500, but less than 15,000 dry metric tons per 365-day period, the monitoring frequency is any one calendar month in each two-month period.
- Any person who prepares sludge and applies to the land greater than or equal to 15,000 dry metric tons per 365-day period, the monitoring frequency is every calendar month.
- The frequency can be less than quarterly only if the activity involves a single source removing sewage sludge for application to the land (or for preparation for application to the land) no more frequently than three times per year and where the total removed is less than 290 dry metric tons per year.

Where a sludge management operation is also a generator subject to SQAR, the more stringent monitoring requirements apply.

Additional monitoring requirements for land application operations may include: (1) Monitoring of parameters (for example, temperature, time and pH) for processes intended to meet pathogen requirements; and (2) Monitoring of parameters (for example, volatile solids, time, temperature, pH and total solids) for processes intended to meet vector attraction reduction requirements. As noted under N.J.A.C. 7:14A-20.7i(5), monitoring of such process parameters "must be performed each day that the process(es) intended to meet any of the requirements is operated and as often each day as necessary".

The applicability of the daily monitoring requirements will vary depending on the amount of sludge produced, the process(es) operated and the frequency of sludge removal. For example, a sludge generator relying on volatile solids reduction to demonstrate vector attraction reduction

could have various monitoring frequencies. If the digester is used in a batch mode, volatile solids testing will only be required prior to digestion and after digestion for each batch. However, if the digester is operated daily on a continuous draw and fill basis, daily volatile solids analyses before and after digestion will be required. Additionally, there may be more than one way for the operator of a process to demonstrate that the process achieves requirements.

There are advantages to monitoring more frequently than is required. For example, if a monitoring event identifies a pollutant limit exceedance, then all sludge used or disposed after the monitoring results are generated will be in violation until another sample is determined to indicate compliance. A person who prepares sludge and samples more frequently than required may be able to document that, in general, the sludge quality consistently meets applicable requirements and that pollutant limit excursions, if any, are abnormal events.

The person who prepares sludge that is disposed in a municipal solid waste landfill must ensure, at a minimum, that the sludge meets the 40 CFR Part 258 requirements for materials disposed in a municipal solid waste landfill. The sludge must not be hazardous (40 CFR Part 258.20) and must pass the paint filter test (40 CFR Part 258.28).

Regardless of the method of sludge use or disposal, all treatment works must have permits that implement applicable technical standards for sludge management. Generally, the permit issued to the treatment works generating the sludge will include applicable sludge quality monitoring as well as other general conditions. This basic information is submitted by compliance with SQAR.

The permit may also include conditions establishing requirements for treatment works that own or operate their own facilities for final use or disposal. Thus, **ALL** sludge preparers (that is, generators as well as persons who manage the sludge) are required to submit information concerning their sludge use or disposal practices.

Chapter 2 References

POTW Sludge Sampling and Analysis Guidance Document. USEPA, Permits Division, Office of Water, Washington D.C. 20460. August 1989.

CHAPTER 3
Sampling Plan

Elements of a Sampling Plan

Department regulations require each sludge generator and preparer to maintain on-site a sampling plan for each parameter required to be monitored under SQAR, or as may be specified in a permit. The general idea is that a sampling plan should include as much information as necessary to ensure consistency from one sampling event to the next to eliminate sampling as the reason for data error.

The key elements of a sampling plan can be divided into four groups focusing on consistency, communication, documentation and data handling:

- Consistency involves the assurance that samples are taken the same way from the same location every sampling event.
- Communication involves making sure the lab understands the proper methods to run, Target Reporting Level and key details regarding the facility.
- Proper sampling activity documentation includes proper sample labeling, chain-of-custody procedures and a logbook of sampling activities.
- Data handling means that after all aspects of the sampling event are documented, the data is reviewed before the data gets submitted.

Sampling is the first, and perhaps the most critical area of the entire process of obtaining sludge quality information. A sample that is representative of the sludge being removed must be acquired in a manner that will not compromise its subsequent analysis. Sampling needs may vary depending upon site location, sample composition, logistics, time of collection, and analytes to be measured. It is also desirable that the final validated procedures are capable of being conducted at a reasonable cost. Sludge ranges from liquids containing less than 1 percent solids to pellets that are greater than 90 percent solids. Hence, a single approach to sampling is neither possible, nor appropriate.

Under SQAR, samples are required to be "collected at locations representative of the chemical and physical characteristics of the sludge removed from the last treatment process before leaving the plant for use or disposal". Proper sampling is an integral part of monitoring the quality of sludge being removed for use or disposal. Information on sludge quality is used by the Department in determining compliance with permit conditions, by generators in developing appropriate sludge management alternatives, and by management sites in determining whether the receipt of a customer sludge is compatible with their chosen sludge use or disposal option. In order to do this, a plan for representative sampling and analysis must be developed. It is required that all sampling procedures be documented in a sampling plan. Some elements that should be documented in a sampling plan include; the sampling points, volumes to be drawn, days and times of collection, required equipment, instructions for labeling samples and ensuring

chain of custody, and a list of contact persons and telephone numbers in case unexpected difficulties arise during sampling. At a minimum, all domestic and industrial treatment works, and sludge management operations are required to maintain a sludge sampling plan on file which meets the following requirements:

1. Identify sludge sampling points at a location which assures homogeneity and best represents the physical and chemical quality of all sludge which is removed from the treatment works for use or disposal.
2. Sampling equipment to be utilized shall be identified and constructed of materials which will not contaminate or react with the sludge (for example, galvanized or zinc coated items may not be used); and
3. The sampling plan shall demonstrate adherence to quality assurance/quality control requirements and procedures for sampling and analysis, including decontamination procedures, consistent with the applicable analytical method.

In addition, site-specific factors must be considered when designing a sampling plan, and the sludge generation and handling process must be understood. For example, is the sludge generated in batches; is there a change in raw materials used in a manufacturing process; can waste composition vary as a function of process temperatures or pressures? Start-up, shut-down, slow-down and maintenance transients can result in the generation of a sludge that is not representative of the normal waste stream. If a sample was unknowingly collected at one of these intervals, incorrect conclusions could be drawn.

Sampling for emerging contaminants create their own set of unique challenges. For example, Per- and Polyfluoroalkyl Substances (PFAS) are ubiquitous in our society. Therefore, there is a much greater chance of cross-contamination and extra precautions need to be taken when designing a sampling plan for PFAS. There are many available sampling guidance documents that have been developed to address sampling for PFAS in sludge. If a sampling plan needs to be addressed to specifically deal with PFAS, the Department would recommend consulting the Biosolids and Sludge PFAS Sampling Guidance that has been developed by the State of Michigan.

Appendix D, “Sampling Plan Template”, provides a template for assistance in development of a sludge sampling plan.

Chapter 3 References

POTW Sludge Sampling and Analysis Guidance Document. USEPA, Permits Division, Office of Water, Washington D.C. 20460. August 1989.

The Wastewater Treatment Plant Operators Guide to Biosolids Sampling Plans. New England Interstate Water Pollution Control Commission, 116 John Street, Lowell, Ma 01852. September 2006.

CHAPTER 4
Sampling Consistency

Selecting Sampling Points

The first key element to ensure that consistent samples are taken is to identify an appropriate sludge sampling point. The sludge sampling point that should be selected is typically the last treatment process the sludge goes through before leaving the treatment plant for use or disposal. For example, if a dewatered sludge off a belt filter press is transported to an incinerator, the sludge coming off the belt filter press should be sampled. If liquid digested sludge is land applied, the sludge should be sampled as it is transferred from the digester to the truck prior to being hauled for land application.

The best place to collect a representative sample is from a place where sludge is "moving" because the sludge is probably mixed well at that point. It is also necessary to consider what the sample is being analyzed for as well as whether it is representative of the sludge that is being removed for use or disposal. It may be appropriate to sample and analyze for certain parameters in different places and at different times. For instance, it may be necessary to determine the volatile solids content of sludge coming from a digester in order to meet vector attraction reduction requirements before dewatering takes place. In this case, it would not be appropriate to analyze for the metal and nutrient content of the liquid digested sludge because the amount of metals and nutrients (and the solids content) could vary significantly after dewatering. Therefore, it would be necessary to perform additional sampling and analysis of the dewatered cake for nutrients, metals and total solids immediately prior to management. If samples are taken from a stationary location, enough grab samples must be taken so as to be representative of the entire area. The greater the quantity of sludge, the greater the number of grab samples needed. These samples can be combined into a composite sample to ensure a representative sample. SQAR (N.J.A.C. 7:14C-1.6(d)3) requires a minimum of five grab samples to form a composite; however, as noted above, more grab samples may need to be taken to form a composite sample that is representative of the sludge removed for use or disposal.

Sample Collection Procedures

Prior to implementing a sampling plan, it is often strategically important to walk through the sampling plan mentally, starting with the preparation of sampling equipment until the time when samples are received at the laboratory. This mental excursion should be in as much detail as can be imagined. By employing this technique, items not included on the equipment list may be discovered, as well as any major oversight that could cause the sampling effort to fail. During this review of the sampling plan, an attempt should be made to anticipate what could go wrong. A solution to anticipated problems should be found, and, if necessary, materials needed for solving these problems should be added to the equipment list. Proper collection procedures that must be addressed in a Sampling Plan include sample type, sample size, and sample equipment and containers.

Sample Type: Grab or composite samples may be appropriate depending on what the sample is being analyzed for and what the operator thinks is representative. A sample from a lagoon, drying bed, compost pile, or truck must consist of numerous samples collected from various locations in the lagoon, bed, pile, or truck that must be combined to make a representative sample. When analyzing for metals and nutrients, SQAR requires a minimum composite of five grab samples be taken over the period of sludge removal. On the other hand, when analyzing for microbiological parameters (for example, fecal coliform or salmonella), individual grab samples usually are required to be taken and analyzed. **(Permits will often specify whether a grab or composite sample is to be taken; therefore, make sure that any permit that has been issued is consulted before designing a sampling plan.)**

A grab sample is a specific quantity of sludge collected at a specific time and location. A single grab sample can represent sludge quality at the time and place it was collected. Extrapolating the analytical results of a single grab sample to represent an entire stockpile or continuous production is not valid. Grab sampling gains validity as historical data accumulates. One instantaneous data point may not convincingly establish sludge quality, but a database showing a consistent pattern may accurately depict sludge quality over time. For continuous processes, improving the comparability of the grab sampling data requires that equally sized samples are collected from the same location. The timing of the grab sample collection should be somewhat random to reflect temporal changes in the sludge. Samples to be submitted for microbial analyses are normally taken as grab samples, so that the time between sample collection and analysis can be documented.

A composite sample is many grab samples that have been collected and mixed together to form a single sample. Grab samples can be randomly collected from a location where sludge is stored, such as a roll-off container or stockpile. In a continuous process, grab samples are typically collected from the same location at a specific time interval over a given period of time. The size of the sample can be weighted to reflect time elapsed or flow. Generally, composite sampling is accomplished by collecting samples of equal size. In the case of continuous processes, the time interval between grab samples is typically kept constant. For example, a 24-hour composite could be obtained by collecting equal size samples every hour from a conveyor moving sludge between dewatering and the hauling vehicle. Data generated from the analyses of a composite sample are only representative of the average sludge quality produced during the time frame over which the sample was collected or of the "batch" that was sampled. As with grab samples, historical data provides the best representation of sludge quality.

In composite sampling, the grab samples that comprise the composite should be completely and thoroughly mixed either by the person performing the sampling or upon receipt in the certified laboratory. During the analysis process only a small portion of the overall sample is taken for analysis. If the composite sample is not thoroughly mixed, the subsample that is removed for analysis may only be representative of a single grab.

Ideally, whether sampling from a container, tank, or pile, several samples should be taken from locations displaced both vertically and horizontally. The number of samples required to be taken must be specified in the sampling plan. At a minimum, a sufficient number and distribution of samples should be taken to address any possible vertical or horizontal anomalies. However, no less than five grab samples must be taken to form the composite sample.

Sample size: Analytical protocols require minimum sample sizes to ensure analytical accuracy and precision. Laboratories should be consulted well in advance of any actual sample collection activities to ascertain the minimum sample size needed for each analytical method.

The amount of sample collected will exceed the amount needed for analysis by a large margin. The sample generally must be reduced to a manageable size for the analyst to handle. Sample size reduction is more difficult for samples for microbial tests because care must be exercised to minimize opportunity for microbial contamination.

For freely flowing liquids, samples can be adequately mixed in the sample bottles by shaking the bottles. There must be room in the bottle for adequate mixing. Compositing of smaller samples is accomplished by pouring them into a larger bottle with adequate freeboard and mixing it by shaking or stirring it thoroughly with a sterile paddle. Pouring off a small part of the contents of a large container into a smaller bottle is a poor procedure, because the top layer of any slurry always contains fewer solids than lower layers. Sampling with a pipette with a wide bore is an acceptable alternative, provided the bore of the pipette is as wide as possible. The sample should be drawn into the pipette slowly and the tip moved through the sample to minimize selective collection of liquid over solid particles.

Sample size reduction for thick sludge is difficult, because shaking is not effective. Stirring with a mechanical mixer or a paddle is often inadequate. A satisfactory approach is to hand mix a composite of any subsamples, and then take a large number of small grabs from the large sample to form the smaller sample for the analyst.

For dry solid samples, the individual particles are frequently large and must be reduced in size to get a representative sample. If the particles are large and a number of subsamples must be combined into a large composite, it may be necessary to reduce the particle size before they are composited. This can be done in a sterile covered chopper, blender, or grinder. The individual subsamples are then combined and mixed by shaking, rotating, and tumbling. A smaller composite is then prepared by combining a number of grabs from all parts of the combined sample. Some other methods used to reduce size, such as "coning and quartering" (ASTM, 1992a) cannot be used for microbiological samples because it is difficult to avoid contaminating the sample when using these procedures.

Sample Equipment and Containers: The most important factors to consider when choosing containers for sludge samples are compatibility with the sludge, cost, resistance to breakage, and volume. Sampling equipment must be constructed of materials which will not contaminate or react with the sludge (for example, galvanized or zinc coated items cannot be used). Thus, it is important to be aware of the potential interactions between sampling equipment and/or container material with analytes of interest. This is true of all sampling equipment used. Containers must not distort, rupture, or leak as a result of chemical reactions with constituents of waste samples. Thus, it is important to have an idea of the properties and composition of the sludge. The containers must have adequate wall thickness to withstand handling during sample collection and transport to the laboratory. Containers with wide mouths are preferred to facilitate transfer of samples from samplers to containers.

Containers for collecting sludge samples are usually made of plastic or glass because these materials are relatively inert and easily cleaned. Glass containers are a good choice but they can

be heavy and may be easily broken. Plastic containers have the advantage of lighter weight; however, they are not suitable for samples subject to analysis for organic compounds because of the potential for sample contamination from phthalate and other hydrocarbons within the plastic or adsorption of the target analyte to the sample container. Glass containers are the best choice for organic constituents, but covers or caps should be lined with Teflon. Sample containers should be filled with care so as to prevent any portion of the collected sample coming in contact with the sampler's gloves, thus causing contamination. Samples should not be collected or stored in the presence of exhaust fumes.

If the samples are to be submitted for analysis of volatile compounds, the samples must be sealed in air-tight containers and filled according to the guidance in *Test methods for Evaluating Solid Waste, Physical/Chemical Methods* (EPA Publication SW-846), Method 5035. Since field preservation is not employed for SQAR samples, it is better to collect a larger volume sample, filling the sample container as full as practical in order to minimize headspace. Such collection procedures generally do not require the collection of a second aliquot, but it still may be adviseable to collect a second sample for screening purposes in order to minimize the loss of volatiles. (See the discussion in Chaper 5 in regards to organic analytes as to why a second aliquot may be necessary.) Sample containers should not be filled near a running motor or any type of exhaust system because discharged fumes and vapors may contaminate the samples. To monitor possible contamination, a trip blank prepared from organic-free reagent water could be carried throughout the sampling, storage, and shipping process.

Before using sampling equipment for the first time and after every use, it must be thoroughly cleaned. Cleaning procedures may differ slightly, depending on the type of sampling equipment and the analysis to be performed. Below is a generalized cleaning procedure that can be used to prepare sampling equipment between sampling events:

1. Rinse equipment with warm tap water to remove the majority of solids.
2. Using a brush and standard low-phosphate lab detergent, scrub the equipment to remove all residues.
3. After scrubbing, triple rinse the equipment with tap water.
4. For the final rinse, triple rinse with deionized water.
5. When sampling for microbial parameters, sterilize the sampling equipment by exposing to high pressure steam of at least 121 degree Celsius for at least 15 minutes.

Ideally, ask your laboratory for certified pre-cleaned, laboratory-grade containers, thus eliminating the need for container cleaning by sample collection staff.

Sample Handling Procedures

Once the sample has been collected, it must be stored and preserved to maintain the chemical and physical properties that it possessed at the time of collection. Appropriate sample containers, preservation and sample holding times are listed in Appendix A. Preservation for sludge samples by the permittee generally consists of cooling and maintaining the samples at the appropriate temperature listed in Appendix A. Although cooling is not a requirement of the analytical methods for most metals, as a practical consideration, since one sample container is generally collected for all analyses, preservation (that is cooling to $\leq$6 degrees C) would then be required. Cooling a sample also greatly reduces the microbial activity that leads to gas production. Gas production can result in enough pressure capable of breaking a glass sample container. For the certified laboratory, samples must be extracted and analyzed within the specified holding times for the results to be considered valid. For composite samples, the holding time starts upon the addition of the last aliquot.

Chapter 4 References

POTW Sludge Sampling and Analysis Guidance Document. USEPA, Permits Division, Office of Water, Washington D.C. 20460. August 1989.

Process Design Manual: Land Application of Sewage Sludge and Domestic Septage, EPA/625/R-95/001. USEPA, Office of Research and Development. September 1995.

Targeted National Sewage Sludge Survey – Sampling and Analysis Technical Report. EPA/822/R-08/016. USEPA, Office of Water, Washington D.C. 20460. January 2009.

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. EPA Publication SW-846 Third Edition (September 1986), Update I (July 1992), Update II (September 1994), Update IIA (August 1993), Update IIB (January 1995), Update III (December 1996), Update IIIA (April 1998), Update IIIB (November 2004) and Update IV (January 2008). Available from the National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161.

The Wastewater Treatment Plant Operators Guide to Biosolids Sampling Plans. New England Interstate Water Pollution Control Commission, 116 John Street, Lowell, Ma 01852. September 2006.

CHAPTER 5
Sampling Documentation

Sample Documentation

Adequate documentation of sludge sampling activities is important for general program quality assurance/quality control and required by most monitoring regulations. Proper sampling activity documentation includes proper sample labeling, chain-of-custody procedures and a logbook of sampling activities.

Sample Labels: Labels and ink should be waterproof, and labels should be fixed to containers with clear waterproof tape. Tape completely around container and over label to prevent accidental label loss or ink smear during shipping and handling. Sample labels should include the following information:

- Sample identification number
- Facility Name (being sampled)
- Date(s), Time(s) of collection
- Sample Location (sample point)
- Type of sample (For example, individual grab, 5-grab composite)
- Collector
- Preservation (for example, cooling procedure)
- Analytical Parameter(s)
- Special Conditions or Remarks

Chain-of-custody: When samples are taken and transported to a lab, it is important to track who is involved and at what time they had possession of the sample. This is called a chain-of-custody record. A chain-of-custody document provides a record of sample transfer from person to person. This document helps protect the integrity of the sample by ensuring that only authorized persons have custody of the sample. In addition, the chain-of-custody procedure ensures an enforceable record of sample transfer which is necessary if the sample results are to be used in a judicial proceeding. This document shall record each sample's collection and handling history from time of collection until analysis as well as the information listed on each sample bottle. All personnel handling the sample shall sign, date and note the time of day on the chain-of-custody document.

A chain-of-custody sheet is usually provided by the lab doing the analysis when providing the sampling kit or containers for shipping. The chain-of-custody should have the following information on it: the sample collector's name, signature of the collector, date and time of

collection, place and address of collection, and the signatures of the persons involved in the chain of possession.

Field Logbook: After the development of the sampling plan, and the collecting of consistent samples in accordance with the sampling plan, the next step is to document all aspects of the sampling event. In other words, keep a field logbook. The details of the sampling event must be documented, including such information as the quantity and type of sludge sampled, location of samples collected, treatment process condition and deviations, and weather conditions. Such documentation is useful if the laboratory results are atypical or suspect, and can serve to demonstrate that the proper sampling process was used. All information pertinent to sampling must be recorded in the field logbook. As required by SQAR, entries in the logbook must include, at a minimum, the following records:

- Date, exact place, and time of sampling or measurement;
- Type of sample collected (composite or grab, number of grab samples, and for continuous processes, the interval between grab samples; for composite samples, the number of grab samples collected and their relative weighting);
- The individual(s) who performed the sampling or measurement; and
- The weather conditions at the time of sampling and other observations which could potentially impact the laboratory analytical results (including deviations from established protocols).

Sampling situations vary widely. Therefore, no general rule can be given as to the extent of information that must be entered in the logbook. A good rule, however, is to record sufficient information so that anyone can reconstruct the sampling without reliance on the collector's memory. Thus, additional information that can be recorded in the logbook includes:

- Number of samples and volume of sample taken;
- Sampling equipment and a brief description of sampling procedures;
- Sample identification number(s);
- Sample transportation method (e.g. name of laboratory, UPS, Federal Express); and
- Signatures of personnel responsible for observations.

Chapter 5 References

POTW Sludge Sampling and Analysis Guidance Document. USEPA, Permits Division, Office of Water, Washington D.C. 20460. August 1989.

Process Design Manual: Land Application of Sewage Sludge and Domestic Septage, EPA/625/R-95/001. USEPA, Office of Research and Development. September 1995.

Targeted National Sewage Sludge Survey – Sampling and Analysis Technical Report. EPA/822/R-08/016. USEPA, Office of Water, Washington D.C. 20460. January 2009.

The Wastewater Treatment Plant Operators Guide to Biosolids Sampling Plans. New England Interstate Water Pollution Control Commission, 116 John Street, Lowell, Ma 01852. September 2006.

CHAPTER 6
Communication

Overview

After documenting the sampling event, the sampling plan objectives need to be clearly communicated with the selected New Jersey Certified Laboratory. Details that should be communicated with the certified laboratory and specified in a contract are:

- Desired or required analytes to be tested (See Chapter 2);
- Analytical methods and protocols required for each test (for example, metals, fecal coliform, nutrients) that will be performed (See Appendix A);
- Clear identification and discussion of Target Reporting Level (See Appendix A); and
- Details about the sludge sample that could potentially impact the analytical results (see below).

Every certified laboratory is required to have a program to validate the quality of its data. With each data report, generators should request the following information:

- A copy of completed sample chain-of-custody records;
- Documentation of the laboratory certification number;
- Documentation of the laboratory method used for each sample analyzed;
- Documentation of sample collection date and time, and analysis date and time;
- Information validating that duplicates, spikes, and standard and blank analyses for samples meet internal QA/QC requirements; and
- Documentation of deviations or anomalies during sample preparation and analysis.

In order to verify that a laboratory's certification is current, generators should annually request that they be provided a copy of the certificate showing the parameters for which the lab is certified. Questions to ask for assistance in selecting a New Jersey Certified Laboratory are available in Appendix C of this Guidance Document.

Choosing the Correct Analytical Procedure

Using the proper laboratory method to analyze sludge is critical (See Appendix A). Sludge generators should review the required methods in order to understand the proper holding times and sample requirements outlined. In addition, a basic understanding of the laboratory method, combined with the understanding of how sludge is produced, can help a technician troubleshoot atypical or suspect data. Generators should also verify that the laboratory is certified for each required method by the State of New Jersey, Department of Environmental Protection, Office of Quality Assurance.

The methods used by the permittee's laboratory or its contract laboratory must be uniform, thus, eliminating methodology as a variable when data from different laboratories is compared. The sludge matrix is more complex and variable than the wastewater matrix; therefore, the laboratory's development of sample preparation techniques is of particular concern. The primary difference in sludge preservation, as opposed to wastewater or drinking water preservation, is that sludge samples generally should not be chemically preserved in the field because the sludge matrix makes it difficult to thoroughly mix the preservative into the sample. Therefore, samples requiring preservation shall be preserved upon receipt in the laboratory which will be conducting the analyses.

Another analytical challenge is that the various treatment processes lead to differences in the moisture content of the final sludge removed for use or disposal. Some facilities produce liquid sludge with a percent solids content ranged from less than 1 percent to about 4 percent. For facilities that produce a more solid sludge, the percent solids content can range from about 5 percent to greater than 90 percent.

Federal regulations at 40 CFR 503.8 require that a representative sample of sewage sludge be collected and analyzed. The purpose of this requirement is to ensure that the samples of sewage sludge that are collected are representative of the sewage sludge that is used or disposed. Analytical methods are specified in 40 CFR 503.8 for enteric viruses, fecal coliform, helminth ova, inorganic pollutants, *Salmonella* sp. bacteria, specific oxygen uptake rate, and total, fixed and volatile solids. Additional analytical methods and appropriate holding times are referenced in 40 CFR Part 136 for fecal coliform and *Salmonella* sp. bacteria. In addition, 40 CFR Part 503 references a document that contains procedures that can be used to calculate the percent volatile solids reduction. This document, *Environmental Regulations and Technology - Control of Pathogens and Vectors*, EPA-625/R-92/013, U.S. Environmental Protection Agency, Washington D.C., 1992, also discusses how to collect samples that are analyzed for pathogens. These method requirements are referenced in Appendix A of this Guidance Document.

In addition, the above analytical requirements are incorporated by reference into New Jersey regulation as part of SQAR. Additional requirements included under SQAR require all analytical testing to be performed by a laboratory certified in New Jersey for the specific method to be utilized. A common analytical error frequently made is that laboratories conduct analyses on sludge using analytical methods developed for water and wastewater. Analytical methods for water and wastewater are found in *Standard Methods*, while the solid waste analytical methods are found in *Test Methods for Evaluating Solid Wastes* (EPA SW-846). For sludge samples, and as required by 40 CFR 503.8 and SQAR, analyses must be conducted using SW-846 methods, when such methods are available.

As stated, Appendix A of this Guidance Document summarizes acceptable procedures for sludge analyses as well as the maximum allowable sample holding times, sample preservation techniques, sample containers, sample preparation methods, and additional comments that may be pertinent to the analytical method. Note that more than one method may be provided for some pollutants. The difference between the methods is usually the equipment used and the level of detection desired. Each of the methods is acceptable to the Department, but certain sample characteristics or Target Reporting Level (discussed later in this chapter) may require one method to be used instead of another.

Many analytical methods applicable to sludge instruct the laboratory to prepare a specific known weight of a solid material for analysis (for example, some methods for organics specify using 30 grams of sample). However, that sample aliquot may contain significant amounts of moisture. Those same methods may treat samples that are pourable liquids as if they contain little or no solids, and specify using a known volume for the analysis although that volume may contain measurable solids as well. These differences in how liquid and solid samples are prepared and analyzed, as well as the differences in the amount of solids or moisture in the two types of samples, mean that any measure of method sensitivity will depend on the initial mass or volume chosen for analysis and its moisture content. To minimize the potential sensitivity differences, laboratories must determine the percentage of solids of each sample first, and then use that information to select a portion of the sample for the analysis that contains the method-specified sample weight or volume on a dry-weight basis.

Inorganic Analytes: After reviewing the methods that will be used to analyze metals in their sludge, generators must make sure the results are reported in milligrams per kilogram (mg/kg) of dry weight (the percent total solids analysis is required in addition to the specified metal analysis). Generators can further help laboratory analysts by providing the limit concentration of each metal. For example, if a generator distributes Class A exceptional quality biosolids, the laboratory should be provided with the list of metal limits in Table 3 of 40 CFR 503.13. Understanding these limits will help the laboratory know when to alert the generator if the results exceed these parameters or, if needed, to prepare dilutions so the resulting data is either a "real" number or the detection limit is less than the metal concentration limit listed. Appendix A as part of this Guidance Document lists desired Target Reporting Level based on appropriate environmental indicators.

As stated above, laboratories must determine the percentage of solids of each sample first, and then use that information to select a portion of the sample for the analysis that contains the method-specified sample weight or volume on a dry-weight basis. For metals (except mercury), *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods* (EPA Publication SW-846), Method 3050 or Method 3051 is the required preparation method using an equivalent to one gram dry weight. This requirement of sample size then proceeds with Method 3050 (or 3051) for sample digestion on a whole sample basis.

This system of using sufficient sample size is practical down to the 1.0 percent solid range. At values below this level, the sample size needed for analysis is too large for analytical processing. Therefore, a maximum sample size of 100 grams is recommended for sludge samples with a percent total solids of one percent or less. This sample size is sufficient to give an accurate sample of the sludge that will allow a reasonable preparation by Method 3050 or Method 3051 while not substantively lessening the analytical quality or quantitative results. (See Table 1 below for sample size recommendations.)

TABLE 1

SAMPLE SIZE RECOMMENDATIONS

Sludge Sample Percent Solids (Determined by Method 2540G)	Sample Size (Wet weight basis)	Approximate volume	Sample size (Dry weight basis)
10 percent	10.0 grams	10 ml	1 gram
5 percent	20.0 grams	20 ml	1 gram
1 percent	100.0 grams	100 ml	1 gram

There are several analytical techniques for trace inorganic analyte determinations. Inductively Coupled Plasma (ICP) is the technique employed most often for the analysis of inorganics in sludge (except for mercury). ICP's primary advantage is that it allows for determination of many elements in a short time. The primary disadvantage is that it is very difficult to find trace concentrations of some inorganics when there is the large presence of a single analyte. For example, in lime treated sludge the large concentration of calcium makes finding other inorganics difficult. In addition, the presence of high amounts of aluminum in a sample, such as for Alum sludge from Potable Water Treatment Systems, could result in a significant false signal for arsenic. Similar problems with interferences arise when ferric chloride as well as other potential polymers are used. As previously stated, it is imperative that the sludge generator provide detailed information to their contract laboratory with regards to these potential interferences in their sludge sample.

Nutrients: The Sludge Quality Assurance Regulations and Federal Regulations at 40 CFR Part 503.8 require that analyses be conducted in accordance with the test procedures in *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods* (EPA Publication SW-846) when such methods are available. Therefore, the required method for Nitrate-Nitrogen is EPA Publication SW-846, Method 9056 or 9210.

EPA Publication SW-846, Method 9056 recommends a 48 hour holding time for Nitrate-Nitrogen. However, longer holding times are appropriate when it can be demonstrated that reported concentrations are not adversely affected from preservation, storage and analysis performed outside the recommended holding times. Nitrate-Nitrogen and Nitrite-Nitrogen can undergo transformation back and forth in environmental samples, with nitrate reduced to nitrite under certain conditions, and nitrite oxidized to nitrate under others. For sludge samples, since nitrite is largely unstable, the process of holding and preparing the sample for analysis is likely to lead to some conversion of nitrite to nitrate. A review of limited data does suggest a slightly

higher concentration for Nitrate-Nitrogen using a 28-day holding time when compared to Nitrate-Nitrogen results from a holding time of 48 hours. Therefore, since both Nitrate and Nitrite are typically found at low levels in sludge, and, from an agronomic perspective, a longer holding time would represent a more conservative result, this Guidance Document allows a longer 28-day holding time for Nitrate-Nitrogen.

The remaining nutrients (for example, Total Kjeldahl Nitrogen and Ammonia Nitrogen) require methods that are found in 40 CFR Part 136 because there is no method for the parameter in EPA Publication SW-846.

pH: The most appropriate method to use for pH measurement is based on the aqueous content of the sample. Sludges which are low in solids (that is <7 percent dry solids) do not require additional fluid to ensure adequate ionization. Therefore, *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods* (EPA Publication SW-846), Method 9040, would be adequate. Under such circumstances, pH measurement can be made directly in the liquid sludge.

If the sludge contains greater than 7 percent dry solids, the sample must first be made into a slurry for the pH measurement. Acceptable procedures for preparing a sample and measuring pH are given in *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods* (EPA Publication SW-846), Method 9045.

Regardless of the method used, pH is very sensitive to temperature, especially at pH 12 and above. Therefore, pH must be measured at 25 degree Celsius or, if measured at a different temperature, converted to an equivalent value at 25 degree Celsius.

Organic Analytes: Gas chromatography, coupled with mass spectrometric detection (GC/MS), is the backbone of the analytical methods for many organic pollutants. Some GC/MS methods include several hundred target analytes, and while full-scan GC/MS is a powerful technique, it involves tradeoffs in sensitivity and selectivity in order to be applied to a large number of analytes simultaneously. Full-scan GC/MS can also be subject to interferences from other materials that are not of interest in the sample.

For volatile organic analysis, the use of methanol as a preservative and extraction solvent introduces a significant dilution factor that will raise the method quantitation limit beyond the operating range of the low concentration direct purge-and-trap procedure. The exact dilution factor will vary, but may make it difficult to meet the Target Reporting Level in Appendix A. Thus, it may be necessary to collect an aliquot for analysis by the closed-system procedure if the Target Reporting Level cannot be achieved, and another aliquot preserved in methanol and analyzed by other procedures.

For polynuclear aromatic hydrocarbons and semivolatile organics, it is beneficial to have every sample extract to be analyzed be subjected to gel-permeation cleanup (GPC) before analysis due to potential interferences. In addition, when a Gas Chromatography / Mass Spectrometry (GC/MS) instrument is operated in full-scan mode, it scans the entire mass range very quickly and there is relatively little time to observe the results at any given mass within that range. This places some practical limits on the sensitivity of the procedure. However, those limitations can be overcome by using the technique known as selected ion monitoring (SIM). To date, many analytes have been reported under SQAR as “not detected” at concentrations greater than the

standards for which subjective comparisons must be made. Based on the successful analysis of extracts using GC/MS with selected ion monitoring in the USEPA's Targeted National Sewage Sludge Survey, the Department recommends the SIM prodecure be employed when necessary to achieve the Target Reporting Level in Appendix A.

Pathogens and indicator organism testing: One of the most important things a generator can do relative to pathogen analysis is to understand the holding and analysis times. Analytical methods for pathogens and indicator organisms often involve short holding times and require several days to complete. Coordinating sample collection times with analysis time is critical to ensure that sludge intended for land application is acceptable at the time of distribution or application. Laboratories should refer to Appendices F through I of the USEPA's *Control of Pathogens and Vector Attraction in Sewage Sludge* for guidance on preparing sludge for pathogen analysis. A generator who observes trends in the pathogen data over time can further enhance the quality of the data by helping the laboratory "fine-tune" sample dilutions to determine the pathogen concentration range of the biosolids data. There are several other factors to note relative to pathogen analyses:

- samples must be collected in sterilized containers;
- data must be reported in dry weight, therefore, a percent total solids analysis is required with each pathogen analysis; and
- 40 CFR 503.32(b)2ii requires that for Class B biosolids seven grab samples be individually analyzed for pathogens, and these results must be averaged using the geometric mean to determine pathogen concentrations.

With regards to holding times for fecal coliform and *salmonella*, 40 CFR Part 136, Table 4 requires that "sample analysis begin immediately, preferably within 2 hours of collection. The maximum transport time to the laboratory is 6 hours, and samples should be processed within 2 hours of receipt at the laboratory. For fecal coliform samples for sewage sludge only, the holding time is extended to 24 hours for the following sample types using either EPA Method 1680 or 1681: Class A compost and Class B aerobically or anaerobically digested."

When analyzing for microbiological parameters, individual grab samples are usually required to be taken and analyzed. However, there are occasions when the collection of a representative sample requires the use of time compositing procedures. For example, if a digester is being sampled during a withdrawal that takes about 15 minutes, a sample can be taken during the first, second, and third 5-minute period. The three separate samples should be brought back to the laboratory and composited into a single sample. Nevertheless, the composite should be prepared within an hour of collecting the first individual grab sample. A longer time period might allow microbial changes to occur in the first sample taken.

Even processed sewage sludge is not inert; in the presence of air it oxidizes slowly. Temperatures can rise to substantial levels. For example, a storage pile of compost or dried sludge may be at room temperature on the outside but could be at 60 degree Celsius at a depth of 2 feet within the pile. The microbiological content of samples from the surface and from the interior of the pile may be considerably different. When there is a large temperature gradient in a storage pile, it is important to include an analysis of the sludge from the cooler section of the pile

where the chance of regrowth of bacterial populations is greatest. In any case, samples from a large pile should be taken at various depths along its length.

All samples for microbial analyses should be cooled to water-ice temperatures when collected or very soon thereafter. For example, enteric viral and bacterial densities are noticeably reduced by even 1 hour of exposure to temperatures of 35 degree Celsius or greater. The requirement of cooling limits the practical size of the sample collection container. A gallon sample bottle will take much longer to cool than a quart bottle. Use of bottles no bigger than a quart is recommended for most samples, particularly if the sludge being sampled is from a process operated at above ambient temperature. For rapid cooling, placing the sample container in a slurry of water and ice produces excellent results. Bagged ice or "blue ice" is effective in maintaining low temperatures but several hours can elapse before this kind of cooling reduces sample temperature to below 10 degree Celsius (Kent and Payne, 1988). The same is true if warm samples are placed in a refrigerator.

The requirement for prompt chilling of samples is appropriate for viruses as well as bacteria. There are fewer laboratories capable of carrying out virus tests than bacteria, so time between analysis and sampling could routinely exceed 24 hours. Fortunately, viruses are not harmed by freezing. Samples can be frozen in a -18 degree Celsius freezer and stored for up to 2 weeks without harm. They then can be packed in dry ice and shipped to the analyzing laboratory.

In the case of wood chips and other large particle size bulking materials, it is appropriate to remove large pieces before analysis takes place. In order to ensure that wood chips are not included in the lab's subsample, the facility should remove wood chips after sampling, being careful not to contaminate, with a sterilized sieve. Alternatively, the laboratory should be asked to remove wood chips from samples before subsampling or analysis is conducted.

Emerging contaminants: As new emerging contaminants are identified and sampling for these new contaminants becomes a requirement for sludge sampling and analysis, the Department will either need to update this guidance document, develop separate specific guidance for each contaminant, or refer to specific guidance developed by other states or organizations. For PFAS, at this time, the Department would recommend consulting the Biosolids and Sludge PFAS Sampling Guidance that has been developed by the State of Michigan.

Identifying and Achieving the Correct Detection Limits

From an analytical standpoint, sludge is a challenging matrix. The concentrations of pollutants present in samples vary depending on the nature of the inputs to the treatment plant. In addition to the pollutants of interest, sludge can contain a number of other components that are potential interferences in the analysis of the pollutants of interest. These components can include naturally occurring materials, as well as materials that may be added to the sludge during processing (e.g. ferric chloride, aluminum sulfate, or lime). These components can manifest themselves as interferences at all stages of the analytical process, from sample preparation through the determinative analysis. To assist the laboratory, generators should explain the sludge generation processes and list all materials added to the sludge so the technicians can resolve any interferences.

Appendix A of this Guidance Document lists all of the parameters required under SQAR, as well as additional parameters required for the land application of sludge. In addition, for most parameters, the Department lists a Target Reporting Level. These targets are based on environmental indicators as well as on the Department's decision as to what is practical. Some targets were driven lower due to the need to provide comparisons between the Department's non-residential soil clean-up standards and sludge intended for land application.

Chapter 6 References

Environmental Regulations and Technology: Control of Pathogens and Vector Attraction in Sewage Sludge. EPA/625/R-92/013. USEPA, Office of research and Development. July 2003.

NPDES Compliance Inspection Manual. EPA/300/B-94/014. USEPA, Enforcement and Compliance Assurance. September 1994.

POTW Sludge Sampling and Analysis Guidance Document. Permits Division, Office of Water, Washington D.C. 20460. August 1989.

Process Design Manual: Land Application of Sewage Sludge and Domestic Septage. EPA/625/R-95/001. USEPA, Office of Research and Development. September 1995.

Targeted National Sewage Sludge Survey – Sampling and Analysis Technical Report. EPA-822-R-08-016. USEPA, Office of Water, Washington D.C. 20460. January 2009.

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. EPA Publication SW-846 Third Edition (September 1986), Update I (July 1992), Update II (September 1994), Update IIA (August 1993), Update IIB (January 1995), Update III (December 1996), Update IIIA (April 1998), Update IIIB (November 2004) and Update IV (January 2008). Available from the National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161.

The Wastewater Treatment Plant Operators Guide to Biosolids Sampling Plans. New England Interstate Water Pollution Control Commission, 116 John Street, Lowell, Ma 01852. September 2006.

Biosolids and Sludge PFAS Sampling Guidance, Michigan Department of Environment, Great Lakes and Energy. November 2019.

CHAPTER 7
Data Handling Procedures

Data Review

Congratulations! By now a sampling plan has been developed, representative samples have been taken, the sampling event has been documented, and the details of the sampling have been communicated to the certified laboratory. However, before the analytical results are submitted, the results must be critically reviewed. Some questions to go over before submitting the results include:

- Is the data believable?
- Are there outliers (high or low) based on historic trends?
- Are the detection levels sufficient?
- Were the correct methods used?
- Are the results in the correct units?

If something doesn't look right, generally, problems with data can be summed up as being associated with one of three things: the sampling event itself, the equipment used or the analytical procedures followed.

The first step to resolving data issues is to go back and review the sampling procedures and the information documented for the sampling event. Of particular importance is the log book: observations of weather, operational problems, anything that may have been observed that was out of the ordinary.

Next, try to rule out equipment as a cause. For example, galvanized sampling lines or containers could cause elevated zinc in the sampling results. In addition, poorly cleaned sampling equipment could potentially bias the results.

Finally, and more difficult to resolve, are problems associated with the analysis. If it is believed that there may be analytical issues, the contract laboratory should be contacted to discuss any concerns, but only after sampling and equipment as a probable cause have been ruled out. In the end, it may be necessary to have the lab run another analysis if an error is suspected or standards are not met. Splitting a sample between two different labs should also be considered if analytical issues or errors are suspected.

Recordkeeping

All domestic and industrial treatment works must retain the documentation specified below for a period of at least five years from the date of the sludge sample. The Department may, for cause, extend the period that the records must be maintained by written notice to the treatment works. Causes for extending the records maintenance period include, for example, enforcement action or

litigation. All domestic and industrial treatment works are required to maintain records of the following:

- The date, exact place, and time of sampling or measurement;
- The individual(s) who performed the sampling or measurement;
- The weather conditions at the time of sampling and other observations which could potentially impact the laboratory analytical results (that is, the log book);
- The date(s) and time(s) laboratory analyses were performed;
- The individual(s) who performed the laboratory analyses;
- The laboratory analytical techniques or methods used;
- The results of such laboratory analyses; and
- The following quality control and quality assurance information:
    - Method blank results;
    - Serial dilution results for metal analyses;
    - Precision and accuracy statement determined by laboratory matrix spikes and matrix spike duplicates on the sludge sample and the quality control sample;
    - Chain-of-custody; and
    - Method detection limits (aqueous matrix and calculated for the sample based on dry weight).

Additional records may be required to be kept depending on the method of sludge use or disposal. Typical records that should be maintained depending on the ultimate management method selected include:

- Data documenting pathogen reduction, including process parameters;
- Data documenting vector attraction reduction (VAR), including process parameters;
- The amount of sludge produced and its final disposition; and
- The amount of outside sludge or septage received, if applicable.

Most data describing the who, what, when, where, and how of your sampling should be retained. Not all of this data has to be reported, but the records should be kept and consulted should the need arise.

Reporting

One potential source of error in reporting is the use of incorrect units. The 40 CFR Part 503 standards and the requirements of SQAR are expressed on a dry weight basis. Pollutant concentrations are reported in dry weight units due to differences in sludge samples. A sludge pollutant concentration reported in dry weight units is a function of the sample's percent solids. Percent solids range from less than one percent to greater than 90 percent. This standardized

reporting unit allows all sludge samples to be evaluated on an equivalent basis with respect to pollutant loads. The laboratory must provide the results on a dry weight basis (that is, mg/kg).

Each domestic or industrial treatment works is required to comply with the land-based sludge management criteria applicable to the ultimate sludge management alternative utilized by the domestic or industrial treatment works. This includes all management sites that are used by the treatment works directly (for example, owner and operator of a sludge incinerator) or indirectly (sludge is hauled to another treatment works which land applies or incinerates their sludge). In determining when to sample, keep in mind the time it takes for shipping, analyzing, and getting back the analysis so a determination of compliance can be made. Each domestic or industrial treatment works has the responsibility to report any noncompliance with the land-based sludge management criteria to the Department. The noncompliance with the land-based sludge management criteria has to be orally reported to the Bureau of Pretreatment and Residuals at (609) 633-3823 and to the ultimate sludge management alternative within 24 hours of the treatment works becoming aware of the noncompliance. A written submission is required within five days thereafter to: Mail Code 401-02B, Chief, Bureau of Pretreatment and Residuals, Division of Water Quality, P.O. Box 420, Trenton, New Jersey 08625, with a copy to the ultimate sludge management alternative. The written submission must include the following information:

1. Dates of occurrence;
2. A description of the noncompliance with the land-based sludge management criteria;
3. The cause of the noncompliance; and
4. Steps being taken to reduce, eliminate and prevent reoccurrence of the noncompliance.

If "not detected" results are above the Target Reporting Level in Appendix A, it may be because the certified laboratory was not aware of the Target Reporting Level requirements. Discuss with your lab in advance possible changes to their preparation procedures to accomplish meeting the Target Reporting Levels.

Finally, after the results have been evaluated, and any issues that have arisen have been satisfied, the results must be submitted on pre-printed reporting forms provided by the Department. Carefully transcribe the results reported by the analytical laboratory onto the pre-printed reporting forms. Double check all of your entries to ensure the correct result is attributed to the appropriate parameter. The Department has prepared a Monitoring Report Form Manual that identifies common reporting mistakes. The MRF Manual also contains information on electronic reporting of data. The MRF Manual may be obtained online at http://www.state.nj.us/dep/dwq/pdf/MRF_Manual.pdf.

Chapter 7 References

Environmental Regulations and Technology: Control of Pathogens and Vector Attraction in Sewage Sludge. EPA/625/R-92/013. USEPA, Office of research and Development. July 2003.

NPDES Compliance Inspection Manual. EPA/300/B-94/014. USEPA, Enforcement and Compliance Assurance. September 1994.

POTW Sludge Sampling and Analysis Guidance Document. Permits Division, Office of Water, Washington D.C. 20460. August 1989.

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. EPA Publication SW-846 Third Edition (September 1986), Update I (July 1992), Update II (September 1994), Update IIA (August 1993), Update IIB (January 1995), Update III (December 1996), Update IIIA (April 1998), Update IIIB (November 2004) and Update IV (January 2008). Available from the National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161.

APPENDIX A

Analytical Parameters, Methods, Holding Times, and Target Reporting Level

Parameter	CAS RN	NJPDES PARAM No.	Analysis Method Description (Note 1)	Regulatory Criteria (mg/kg)	Target Reporting Level (mg/kg) (Note 2)	Maximum Holding Time Sample Preservation (Note 3) Sample Container	Comments Sample Preparation
Table I: Primary Metals and Selected Chemical:							
Total Solids	na	00500	SM 2540 G			7 days Cool ≤6 degree C Plastic or Glass Container	Method 2540 G is the required procedure pursuant to 40 CFR 503.8
Arsenic	7440-38-2	01003	ICP SW-846 Method 6010 ICP/MS SW-846 Method 6020 AA Gaseous Hydride SW-846 Method 7061 AA Graphite Furnace SW-846 Method 7010	19.0 Note 4 41.0 Note 5 10.0 Note 6	10.0[c]	6 months Plastic or Glass Container	See Note 7
Beryllium	7440-41-7	61524	ICP SW-846 Method 6010 ICP/MS SW-846 Method 6020 AA Direct Aspiration SW-846 Method 7000 AA Graphite Furnace SW 846 Method 7010	16.0 Note 4 5.0 Note 6	5.0[c]	6 months Plastic or Glass Container	See Note 7
Cadmium	7440-43-9	61527	ICP SW-846 Method 6010 ICP/MS SW-846 Method 6020 AA Direct Aspiration SW-846 Method 7000 AA Graphite Furnace SW 846 Method 7010	78.0 Note 4 39.0 Note 5 10.0 Note 6	4.7[b]	6 months Plastic or Glass Container	See Note 7

Calcium	7440-70-2	00917	ICP SW-846 Method 6010 AA Direct Aspiration SW-846 Method 7000			6 months Plastic or Glass Container	See Note 7
Chromium	7440-47-3	78473	ICP SW-846 Method 6010 ICP/MS SW-846 Method 6020 AA Direct Aspiration SW-846 Method 7000 AA Graphite Furnace SW 846 Method 7010	70.0 Note 6	20.0[a]	6 months Plastic or Glass Container	See Note 7
Copper	7440-50-8	46394	ICP SW-846 Method 6010 ICP/MS SW-846 Method 6020 AA Direct Aspiration SW-846 Method 7000 AA Graphite Furnace SW 846 Method 7010	3100 Note 4 1500 Note 5	20.0[a]	6 months Plastic or Glass Container	See Note 7
Lead	7439-92-1	78468	ICP SW-846 Method 6010 ICP/MS SW-846 Method 6020 AA Direct Aspiration SW-846 Method 7000 AA Graphite Furnace SW 846 Method 7010	400 Note 4 300 Note 5 110 Note 6	20.0[a]	6 months Plastic or Glass Container	See Note 7
Mercury	7439-97-6	78471	Cold Vapor (manual) SW-846 Method 7471	23.0 Note 4 17.0 Note 5 5.0 Note 6	2.0[a]	28 days Cool ≤6 degree C Plastic or Glass Container	SW-846 Method 7471 applies to mercury in solid or semi-solid wastes. Samples need to be digested prior to analysis. The digestion procedure is contained in the analytical method.
Molybdenum	7439-98-7	78465	ICP SW-846 Method 6010 AA Direct Aspiration SW-846 Method 7000 AA Graphite Furnace SW 846 Method 7010	75.0 Note 5	18.0[b]	6 months Plastic or Glass Container	See Note 7

Nickel	7440-02-0	78469	ICP SW-846 Method 6010 ICP/MS SW-846 Method 6020 AA Direct Aspiration SW-846 Method 7000 AA Graphite Furnace SW 846 Method 7010	1600 Note 4 420 Note 5 70.0 Note 6	20.0[a]	6 months Plastic or Glass Container	See Note 7
Potassium	7440-09-7	78472	ICP SW-846 Method 6010 AA Direct Aspiration SW-846 Method 7000			6 months Plastic or Glass Container	See Note 7
Selenium	7782-49-2	01148	ICP SW-846 Method 6010 ICP/MS SW-846 Method 6020 AA Gaseous Hydride SW-846 Method 7741 AA Graphite Furnace SW 846 Method 7010	390 Note 4 100 Note 5	18.0[b]	6 months Plastic or Glass Container	See Note 7
Zinc	7440-66-6	78467	ICP SW-846 Method 6010 ICP/MS SW-846 Method 6020 AA Direct Aspiration SW-846 Method 7000 AA Graphite Furnace SW 846 Method 7010	23000 Note 4 2800 Note 5	20.0[a]	6 months Plastic or Glass Container	See Note 7
Phosphorus, Total	7723-14-0	78478	Persulfate Digestion SM 4500-P B(5) Followed by Manual: SM 4500-P E EPA Method 365.3 Or Automated ascorbic acid reduction SM 4500-P F or G or H EPA Method 365.1 Semi-auto block digestor EPA Method 365.4	Various Note 5		28 days Cool <6 degree C Plastic or Glass Container	Pay close attention to sample preparation requirements found in section 4500-P B.

Nitrogen, Total Kjeldahl		49579	SM 4500-N_{org} B or C EPA Method 351.1 (TKN) EPA Method 351.2 (TKN) SM 4500-N_{org} D	Various Note 5		28 days Cool ≤6 degree C Plastic or Glass Container	Total kjeldahl nitrogen is the sum of the organic and ammonia nitrogen (N_{org} + NH_3) in a sample. See below for the Standard Methods ammonia portion analysis. Sample digestion and distillation are required and are included or referenced in the method.
Nitrogen, Ammonia		82294	Manual Distillation SM 4500-NH_3 B EPA Method 350.1 Followed by: SM 4500-NH_3 C SM 4500-NH_3 D or E SM 4500-NH_3 F-1997 SM 4500-NH_3 G or H EPA Method 350.1	Various Note 5		28 days Cool ≤6 degree C Plastic or Glass Container	All samples must be distilled using procedure SM-4500-NH3 B or EPA Method 350.1 prior to analysis by one of the specific analysis procedures listed.
Nitrogen, Nitrate		00621	SW-846 Method 9056 SW-846 Method 9210	Various Note 5	500[b]	28 days Note 18 Cool ≤6 degree C Plastic or Glass Container	Nitrate nitrogen is the fully oxidized state of nitrogen. Organics may interfere with Method.
Table IA: Special SQAR Table 1 and Selected Chemical:							
Total Volatile Solids		70322	SM 2540G	Various Note 5		7 days Cool ≤6 degree C Plastic or Glass Container	Method 2540 G is the required procedure pursuant to 40 CFR 503.8
pH		00400	SW-846 Method 9045 SW-846 Method 9040	Various Note 5		Immediate (within 15 minutes for a permit limited parameter; "as soon as possible" for a monitor-only parameter) No preservation Plastic or Glass Container	Method 9045 must be used for samples with >7% solids. Method 9040 may be used for samples with ≤7% solids. pH must be measured in a slurry at 25°C or converted using: pH correction = (0.03 pH units/1.0°C) X (Temp°C_{meas} – 25°C)

Temperature (continuous or grab)		00010	SM 2550B	Various Note 5		Immediate	Whether for VAR or pathogen reduction, monthly calibration is necessary unless a facility has a frequency of monitoring that is less than monthly (for example, quarterly or annually… in that case calibration at least once during the monitoring cycle would suffice.)
Dioxin and Dioxin-like compounds (See Appendix B)	Various	Various	EPA Method 1613		5 ng/kg[d]	Samples extracted within 14 days and extracts analyzed within 40 days following extraction. Cool ≤6 degree C Glass container w/ Teflon-lined lid	
PCBs, Total (209 Congeners)	Various	Various	EPA Method 1668	0.2 Note 4 4.6 Note 8	1.0 ug/kg[d]	Samples extracted within 14 days and extracts analyzed within 40 days following extraction. Cool ≤6 degree C Glass container w/ Teflon-lined lid	
Radium - 226	7440-14-4	09501	SW-846 Method 9315 SW-846 Method 9320	3.0 pCi/g Note 4	0.5 pCi/g[a]	Note 10 6 months Plastic or Glass Container	
Radium - 228	7440-14-4	11501	SW-846 Method 9320		0.5 pCi/g[a]	Note 10 6 months Plastic or Glass Container	
Gamma Spectroscopy	Note 9	Note 9	EML GA-01-R Note 9		0.5 pCi/g[a]	Note 10 6 months Plastic or Glass Container	

Parameter	CAS RN	NJPDES PARAM No.	Analysis Method Description (Note 1)	Regulatory Criteria (mg/kg)	Target Reporting Level (mg/kg) (Note 2)	Maximum Holding Time Sample Preservation (Note 3) Sample Container	Comments Sample Preparation
Salmonella		71204	Analysis Method SM 9260 D EPA Method 1682 Note 11,12 "Control of Pathogens and Vector Attraction in Sewage Sludge", Appendix G (Kenner and Clark), EPA/625/R-92/013, July 2003 Sample Preparation "Control of Pathogens and Vector Attraction in Sewage Sludge", Appendix F, EPA/625/R-92/013, July 2003	<3 per four grams of TS (dry weight) Note 5		Cool <10 degree Celsius (do not freeze) Samples analysis should begin immediately, preferably within 2 hours of collection. The maximum transport time to laboratory is 6 hours, and samples should be processed within 2 hours of receipt at the laboratory. PA Note 14 or Glass container	Takes 3-4 days. Large sample volumes are needed because of the low concentration of *Salmonella* in sludge. Also, due to the large number of *Salmonella* species, more than one procedure may be necessary to adequately determine the presence of *Salmonella*.
Fecal Coliform		74055	Analysis Method SM-9221 C E EPA Method 1680 Note 15,12 EPA Method 1681 Note 16,12 Sample Preparation "Control of Pathogens and Vector Attraction in Sewage Sludge", Appendix F, EPA/625/R-92/013, July 2003	<1000 per gram of TS (dry weight Class A) Note 5 -or- <2,000,000 per gram of total solids (dry weight Class B) Note 5		Cool <10 degree Celsius (do not freeze) Samples analysis should begin immediately, preferably within 2 hours of collection. The maximum transport time to laboratory is 6 hours, and samples should be processed within 2 hours of receipt at the laboratory Note 13. PA Note 14 or Glass container	Analysis takes 24 hours. Procedures very temperature sensitive. MPN procedure is required for analysis of Class A biosolids and recommended for Class B biosolids. Standard Method 9222 D is allowable for Class B biosolids only. Note 17
Specific Oxygen Uptake Rate			SM-2710B, and "Control of Pathogens and Vector Attraction in Sewage Sludge", Appendix D, EPA/625/R-92/013, July 2003	See comments		Sludge must be aerobically digested in the 10-30 degree Celsius range, maintained, and samples processed within 2 hours	Limit is < 1.5 mg of oxygen per hour per gram of total solids (dry weight basis) at a temperature of 20 degrees Celsius.

Enteric Viruses			"American Society for Testing and Materials", Method D4994-89 and "Control of Pathogens and Vector Attraction in Sewage Sludge", Appendix H, EPA/625/R-92/013, July 2003	<1 Plaque-forming Unit per four grams of total solids (dry weight) Note 5		Within 24 hours, Cool ≤ 6 degrees Celsius (up to 2 weeks if frozen ≤ -18 degrees Celsius) (≤ -70 degree C if samples will be stored for more than 2 weeks)	
Helminth Ova			Yanko (1987) and "Control of Pathogens and Vector Attraction in Sewage Sludge", Appendix I, EPA/625/R-92/013, July 2003	<1 per four grams of total solids (dry weight) Note 5		1 month Cool ≤ 6 degrees Celsius (do not freeze)	
Water Extractable Phosphorus			Universal Water Extractable P Test for Manure and Biosolids (adapted from Kleinman et al. 2007)			3 weeks Cool ≤ 6 degrees Celsius Plastic or Glass Container	
Table II, Additional Miscellaneous							
Antimony	7440-36-0	01098	ICP SW-846 Method 6010 ICP/MS SW-846 Method 6020 AA Direct Aspiration SW-846 Method 7000 AA Graphite Furnace SW-846 Method 7010	31.0 Note 4	14.0[b]	6 months Plastic or Glass Container	See Note 7

Silver	7440-22-4	01078	ICP SW-846 Method 6010 ICP/MS SW-846 Method 6020 AA Direct Aspiration SW-846 Method 7000 AA Graphite Furnace SW-846 Method 7010	390 Note 4	10.0[a]	6 months Plastic or Glass Container	See Note 7
Thallium	7440-28-0	34480	ICP SW-846 Method 6010 ICP/MS SW-846 Method 6020 AA Direct Aspiration SW-846 Method 7840* SW-846 Method 7000 AA Graphite Furnace SW-846 Method 7841* SW-846 Method 7010		5.0[c]	6 months Plastic or Glass Container	See Note 7
Cyanide	57-12-5	00721	Distillation SW-846 Method 9010 Determinative SW-846 Method 9213 SW-846 Method 9014	47.0 Note 4	47.0[c]	14 days Cool ≤6 degree C Plastic or Glass Container	
Table III, Volatile Organic Compounds			Analysis Procedure SW-846 Method 8260 Extraction Procedure Purge and Trap SW- 846 Method 5035			14 days Cool ≤6 degree C Glass Container with Teflon-lined lids See individual methods for preservative requirements	Method used to quantify most volatile organic compounds that have boiling points below 200°C and that are insoluble or slightly soluble in water. The laboratory where volatiles analysis is performed should be completely free of solvents. Extraction procedure is dependent on acceptable detection limits.
Acrolein	107-02-08	34213	SW-846 Method 8260	0.5 Note 4	0.5[c]		
Acrylonitrile	107-13-1	34218	SW-846 Method 8260	0.9 Note 4	0.9[c]		
Benzene	71-43-2	34237	SW-846 Method 8260	2.0 Note 4	2.0[c]		

Bromoform	75-25-2	34290	SW-846 Method 8260	81.0 Note 4	10.0[b]		
Carbon Tetrachloride	56-23-5	34299	SW-846 Method 8260	2.0 Note 4	2.0[c]		
Chlorobenzene	108-90-7	34304	SW-846 Method 8260	510 Note 4	10.0[b]		
Chlorodibromomethane	124-48-1	34309	SW-846 Method 8260	3.0 Note 4	3.0[c]		
Chloroethane	75-00-3	34314	SW-846 Method 8260	220 Note 4	10.0[b]		
2-Chloroethylvinyl Ether	110-75-8	34579	SW-846 Method 8260		15.0[b]		
Chloroform	67-66-3	34318	SW-846 Method 8260	0.6 Note 4	0.6[c]		
Dichlorobromomethane	75-27-4	34330	SW-846 Method 8260	1.0 Note 4	1.0[c]		
1,1-Dichloroethane	75-34-3	34499	SW-846 Method 8260	8.0 Note 4	8.0[c]		
1,2-Dichloroethane	107-06-2	34534	SW-846 Method 8260	0.9 Note 4	0.9[c]		
1,1-Dichloroethylene	75-35-4	34504	SW-846 Method 8260	11.0 Note 4	11.0[c]		
1,2-Dichloropropane	78-87-5	34544	SW-846 Method 8260	2.0 Note 4	2.0[c]		
trans-1,3-Dichloropropene	10061-02-6	73404	SW-846 Method 8260	2.0 Note 4	2.0[c]		
Ethylbenzene	100-41-4	34374	SW-846 Method 8260	7800 Note 4	11.0[b]		
Methyl Bromide	74-83-9	34416	SW-846 Method 8260	25.0 Note 4	12.0[b]		
Methyl Chloride	74-87-3	34421	SW-846 Method 8260	4.0 Note 4	4.0[c]		
Methylene Chloride	75-09-2	34426	SW-846 Method 8260	46.0 Note 4	10.0[b]		
1,1,2,2-Tetrachloroethane	79-34-5	34519	SW-846 Method 8260	1.0 Note 4	1.0[c]		

Tetrachloroethylene	127-18-4	34478	SW-846 Method 8260	43.0 Note 4	10.0[b]		
Toluene	108-88-3	34483	SW-846 Method 8260	6300 Note 4	12.0[b]		
1,2-trans-Dichloroethylene	156-60-5	34549	SW-846 Method 8260	300 Note 4	10.0[b]		
1,1,1-Trichloroethane	71-55-6	34509	SW-846 Method 8260	160000 Note 4	10.0[b]		
1,1,2-Trichloroethane	79-00-5	34514	SW-846 Method 8260	2.0 Note 4	2.0[c]		
Trichloroethylene	79-01-6	34487	SW-846 Method 8260	3.0 Note 4 10000 Note 8	3.0[c]		
Vinyl Chloride	75-01-4	34495	SW-846 Method 8260	0.7 Note 4	0.7[c]		
Table IV, Acid-extractable:			Analysis Procedure SW-846 Method 8270 Extraction Procedure See SW-846 Method 3500 for discussion on appropriate techniques Clean-up Procedure See SW-846 Method 3600 for discussion on appropriate techniques			Samples extracted within 14 days and extracts analyzed within 40 days following extraction Cool to ≤6 degree C Glass container with Teflon-lined lid	Method is used to quantify most B/N/A organic compounds that are soluble in methylene chloride. Such compounds include polynuclear aromatic hydrocarbons and pesticides, phthalate esters, ketones, anilines, pyridines, quinolines, aromatic nitro compounds and phenols. Extraction procedure is dependent on acceptable detection limits.
2-Chlorophenol	95-57-8	34589	SW-846 Method 8270	310 Note 4	19.0[a]		
2,4-Dichlorophenol	120-83-2	34604	SW-846 Method 8270	180 Note 4	19.0[a]		
2,4-Dimethylphenol	105-67-9	34609	SW-846 Method 8270	1200 Note 4	19.0[a]		
4,6-Dinitro-o-cresol	534-52-1	79533	SW-846 Method 8270	6.0 Note 4	6.0[c]		
2,4 Dinitrophenol	51-28-5	34619	SW-846 Method 8270	120 Note 4	19.0[a]		
2-Nitrophenol	88-75-5	34594	SW-846 Method 8270		19.0[a]		
4-Nitrophenol	100-02-7	34649	SW-846 Method 8270		19.0[a]		

p-chloro-m-cresol	59-50-7	34455	SW-846 Method 8270		19.0[a]		
Pentachlorophenol	87-86-5	39061	SW-846 Method 8270	0.9 Note 4	0.9[c]		
Phenol, Single Compound	108-95-2	34695	SW-846 Method 8270	18000 Note 4	19.0[a]		
2,4,6 Trichlorophenol	88-06-2	34624	SW-846 Method 8270	19.0 Note 4	19.0[c]		
Table V, Base-Neutral:			Analysis Procedure SW-846 Method 8270 Extraction Procedure See SW-846 Method 3500 for discussion on appropriate techniques Clean-up Procedure See SW-846 Method 3600 for discussion on appropriate techniques			Samples extracted within 14 days and extracts analyzed within 40 days following extraction Cool to ≤6 degree C Glass container with Teflon-lined lid	Method is used to quantify most B/N/A organic compounds that are soluble in methylene chloride. Such compounds include polynuclear aromatic hydrocarbons and pesticides, phthalate esters, ketones, anilines, pyridines, quinolines, aromatic nitro compounds and phenols. Extraction procedure is dependent on acceptable detection limits.
Acenaphthene	83-32-9	34208	SW-846 Method 8270	3400 Note 4	20.0[a]		
Acenaphthylene	208-96-8	34203	SW-846 Method 8270	300000 Note 4	20.0[a]		
Anthracene	120-12-7	34223	SW-846 Method 8270	17000 Note 4	20.0[a]		
Benzidine	92-87-5	39121	SW-846 Method 8270	0.7 Note 4	0.7[c]		
Benzo(a)anthracene	56-55-3	34529	SW-846 Method 8270	5.0 Note 4	5.0[c]		
Benzo(a)pyrene	50-32-8	34250	SW-846 Method 8270	0.5 Note 4 15.0 Note 8	0.5[c]		
3,4 Benzofluoranthene, (Benzo(b)fluoranthene)	205-99-2	79531	SW-846 Method 8270	5.0 Note 4	5.0[c]		
Benzo(ghi)perylene	191-24-2	34524	SW-846 Method 8270	30000 Note 4	20.0[a]		
Benzo(k)fluoranthene	207-08-9	34245	SW-846 Method 8270	45.0 Note 4	20.0[a]		
Bis(2-chloroethoxy) methane	111-91-1	34281	SW-846 Method 8270		20.0[a]		

Bis(2-chloroethyl) ether	111-44-4	34276	SW-846 Method 8270	0.4 Note 4	0.4[c]		
Bis(2-chloroisopropyl) ether	108-60-1	34286	SW-846 Method 8270	23.0 Note 4	20.0[a]		
Bis(2-ethylhexyl) phthalate	117-81-7	39102	SW-846 Method 8270	35.0 Note 4	20.0[a]		
4-Bromophenyl phenyl ether	101-55-3	34639	SW-846 Method 8270		20.0[a]		
Butyl benzyl phthalate	85-68-7	34295	SW-846 Method 8270	1200 Note 4	20.0[a]		
2-Chloronaphthalene	91-58-7	34584	SW-846 Method 8270		20.0[a]		
4-Chlorophenyl phenyl ether	7005-72-3	34644	SW-846 Method 8270		20.0[a]		
Chrysene	218-01-9	34323	SW-846 Method 8270	450.0 Note 4	20.0[a]		
Dibenzo(a,h)anthracene	53-70-3	34559	SW-846 Method 8270	0.5 Note 4	0.5[c]		
1,2-Dichlorobenzene	95-50-1	34539	SW-846 Method 8270 SW-846 Method 8260	5300 Note 4	20.0[a]		
1,3-Dichlorobenzene	541-73-1	34569	SW-846 Method 8270 SW-846 Method 8260	5300 Note 4	20.0[a]		
1,4-Dichlorobenzene	106-46-7	34574	SW-846 Method 8270 SW-846 Method 8260	5.0 Note 4	5.0[c]		
3,3-Dichlorobenzidine	91-94-1	34634	SW-846 Method 8270	1.0 Note 4	1.0[c]		
Diethyl phthalate	84-66-2	34339	SW-846 Method 8270	49000 Note 4	20.0[a]		
Dimethyl phthalate	131-11-3	34344	SW-846 Method 8270		20.0[a]		
Di-n-butyl phthalate	84-74-2	39112	SW-846 Method 8270	6100 Note 4	20.0[a]		
2,4-Dinitrotoluene	121-14-2	34614	SW-846 Method 8270	0.7 Note 4	0.7[c]		
2,6-Dinitrotoluene	606-20-2	34629	SW-846 Method 8270	0.7 Note 4	0.7[c]		
Di-n-octyl Phthalate	117-84-0	34599	SW-846 Method 8270	2400 Note 4	20.0[a]		

1,2-Diphenylhydrazine	122-66-7	34349	SW-846 Method 8270	0.7 Note 4	0.7[c]		
Fluoranthene	206-44-0	34379	SW-846 Method 8270	2300 Note 4	20.0[a]		
Fluorene	86-73-7	34384	SW-846 Method 8270	2300 Note 4	20.0[a]		
Hexachlorobenzene	118-74-1	39701	SW-846 Method 8270	0.3 Note 4 29.0 Note 8	0.3[c]		
Hexachlorobutadiene, (Hexachloro-1,3-butadiene)	87-68-3	39705	SW-846 Method 8270 SW-846 Method 8260	6.0 Note 4 600 Note 8	6.0[c]		
Hexachlorocyclo-pentadiene	77-47-4	34389	SW-846 Method 8270	45.0 Note 4	20.0[a]		
Hexachloroethane	67-72-1	34399	SW-846 Method 8270 SW-846 Method 8260	12.0 Note 4	12.0[c]		
Indeno(1,2,3-cd)pyrene	193-39-5	34406	SW-846 Method 8270	5.0 Note 4	5.0[c]		
Isophorone	78-59-1	34411	SW-846 Method 8270	510 Note 4	20.0[a]		
Naphthalene	91-20-3	34445	SW-846 Method 8270 SW-846 Method 8260	6.0 Note 4	6.0[c]		
Nitrobenzene	98-95-3	34450	SW-846 Method 8270 SW-846 Method 8260	5.0 Note 4	5.0[c]		
N-nitrosodimethylamine	62-75-9	34441	SW-846 Method 8270	0.7 Note 4 2.1 Note 8	0.7[c]		
N-nitrosodi-n-propylamine	621-64-7	34431	SW-846 Method 8270	0.2 Note 4	0.2[c]		
N-nitrosodiphenylamine	86-30-6	34436	SW-846 Method 8270	99.0 Note 4	20.0[a]		
Phenanthrene	85-01-8	34464	SW-846 Method 8270	300000 Note 4	20.0[a]		
Pyrene	129-00-0	34472	SW-846 Method 8270	1700 Note 4	20.0[a]		
1,2,4-Trichlorobenzene	120-82-1	34554	SW-846 Method 8270 SW-846 Method 8260	73.0 Note 4	20.0[a]		

Table VI, Pesticides and PCBs:			Analysis Procedure SW-846 Method 8081 SW-846 Method 8082 Extraction Procedure See SW-846 Method 3500 Clean-up Procedure See SW-846 Method 3600			Samples extracted within 14 days and extracts analyzed within 40 days following extraction Cool to ≤6 degree C Glass container with Teflon-lined lid	Both the pesticides and PCBs are bioaccumulative, stable and toxic. Phthalate esters can pose a major interference problem when using an EC detector. Extraction method dependent on acceptable detection limits.
Aldrin	309-00-2	39333	SW-846 Method 8081	0.04 Note 4 2.7 Note 8	0.04[c]		
Alpha BHC (Alpha HCH)	319-84-6	73364	SW-846 Method 8081	0.1 Note 4	0.1[c]		
Beta BHC (Beta HCH)	319-85-7	73365	SW-846 Method 8081	0.4 Note 4	0.4[c]		
Gamma-BHC (Lindane)	58-89-9	39343	SW-846 Method 8081	0.4 Note 4 84.0 Note 8	0.4[c]		
BHC Delta	319-86-8	34262	SW-846 Method 8081		7.0[b]		
Chlordane (Tech Mix & Metab.)	(see note 4)	39351	SW-846 Method 8081	0.2 Note 4 86.0 Note 8	0.2[c]		
4,4'-DDT	50-29-3	39301	SW-846 Method 8081	2.0 Note 4 120Note 8	2.0[c]		
4,4'-DDD	72-54-8	39311	SW-846 Method 8081	3.0 Note 4	3.0[c]		
4,4'-DDE	72-55-9	39321	SW-846 Method 8081	2.0 Note 4	2.0[c]		
Dieldrin	60-57-1	39383	SW-846 Method 8081	0.04 Note 4 2.7 Note 8	0.04[c]		
Alpha Endosulfan	959-98-8	34361	SW-846 Method 8081	470 Note 4	7.0[b]		
Beta Endosulfan	33213-65-9	34356	SW-846 Method 8081	470 Note 4	7.0[b]		
Endosulfan Sulfate	1031-07-8	34354	SW-846 Method 8081	470 Note 4	7.0[b]		
Endrin	72-20-8	75048	SW-846 Method 8081	23.0 Note 4	8.0[b]		
Endrin Aldehyde	7421-93-4	34369	SW-846 Method 8081		7.0[b]		

Heptachlor	76-44-8	75044	SW-846 Method 8081	0.1 Note 4 7.4 Note 8	0.1[c]		
Heptachlor Epoxide	1024-57-3	39423	SW-846 Method 8081	0.07 Note 4	0.07[c]		
PCB-1242	53469-21-9	39499	SW-846 Method 8082	0.2 Note 4a 4.6 Note 8a	0.2[c]		
PCB-1254	11097-69-1	39507	SW-846 Method 8082	0.2 Note 4a 4.6 Note 8a	0.2[c]		
PCB-1221	11104-28-2	73155	SW-846 Method 8082	0.2 Note 4a 4.6 Note 8a	0.2[c]		
PCB-1232	11141-16-5	39495	SW-846 Method 8082	0.2 Note 4a 4.6 Note 8a	0.2[c]		
PCB-1248	12672-29-6	39503	SW-846 Method 8082	0.2 Note 4a 4.6 Note 8a	0.2[c]		
PCB-1260	11096-82-5	39511	SW-846 Method 8082	0.2 Note 4a 4.6 Note 8a	0.2[c]		
PCB-1016	12674-11-2	39514	SW-846 Method 8082	0.2 Note 4a 4.6 Note 8a	0.2[c]		
Toxaphene	8001-35-2	39403	SW-846 Method 8081	0.6 Note 4 10.0 Note 8	0.6[c]		
Table VII, Conventional and Nonconventional:							
Aluminum, Total	7429-90-5	01105	ICP SW-846 Method 6010 ICP/MS SW-846 Method 6020 AA Direct Aspiration SW-846 Method 7000	78000 Note 4	1000[a]	6 months Plastic or Glass Container	See Note 7
Barium, Total	7440-39-3	01008	ICP SW-846 Method 6010 ICP/MS SW-846 Method 6020 AA Direct Aspiration SW-846 Method 7000 AA Graphite Furnace SW-846 Method 7010	16000 Note 4	100[a]	6 months Plastic or Glass Container	See Note 7
Boron, Total	7440-42-8	01022	ICP SW-846 Method 6010		20.0[a]	6 months Plastic or Glass Container	See Note 7

Cobalt, Total	7440-48-4	01037	ICP SW-846 Method 6010 ICP/MS SW-846 Method 6020 AA Direct Aspiration SW-846 Method 7000 AA Graphite Furnace SW-846 Method 7010	590 Note 4	20.0[a]	6 months Plastic or Glass Container	See Note 7
Iron, Dry Weight	7439-89-6	78474	ICP SW-846 Method 6010 AA Direct Aspiration SW-846 Method 7000 AA Graphite Furnace SW-846 Method 7010			6 months Plastic or Glass Container	See Note 7
Magnesium, Dry Weight	7439-95-4	00924	ICP SW-846 Method 6010 AA Direct Aspiration SW-846 Method 7000		1000[a]	6 months Plastic or Glass Container	See Note 7
Manganese, Total	7439-96-5	01055	ICP SW-846 Method 6010 ICP/MS SW-846 Method 6020 AA Direct Aspiration SW-846 Method 7000 AA Graphite Furnace SW-846 Method 7010	5900 Note 4	20.0[a]	6 months Plastic or Glass Container	See Note 7
Strontium, Total	7440-24-6	01082	ICP SW-846 Method 6010 AA Direct Aspiration SW-846 Method 7000		20.0[a]	6 months Plastic or Glass Container	See Note 7
Tin, Total	7440-31-5	01102	ICP SW-846 Method 6010 AA Direct Aspiration SW-846 Method 7000		10.0[b]	6 months Plastic or Glass Container	See Note 7
Titanium, Total	7440-32-6	01152	ICP SW-846 Method 6010		20.0[a]	6 months Plastic or Glass Container	See Note 7

Vanadium, Total	7440-62-2	01087	ICP SW-846 Method 6010 AA Direct Aspiration SW-846 Method 7000 AA Graphite Furnace SW-846 Method 7010	78.0 Note 4	20.0[a]	6 months Plastic or Glass Container	See Note 7
Zirconium, Total	7440-67-7	01162	ICP SW-846 Method 6010			6 months Plastic or Glass Container	See Note 7
Table VIII, Hazardous Substances:							
Acetone	67-64-1	81552	SW-846 Method 8260	70000 Note 4	2.0[b]	See Table III above	
Acetonitrile (Methyl cyanide)	75-05-8	76997	SW-846 Method 8260		2.0[b]	See Table III above	
Acetophenone	98-86-2	73272	SW-846 Method 8270	2.0 Note 4	2.0[c]	See Table V above	
2-Acetylaminofluorene (2-AFF)	53-96-3	82204	SW-846 Method 8270		2.0[b]	See Table V above	
Acrylamide	79-06-1	50796	SW-846 Method 8032 SW-846 Method 8316		0.5[a]	Samples extracted and analyzed within 7 days Cool ≤ 6 degree C Glass with Teflon-lined lid	
Allyl chloride	107-05-1	04385	SW-846 Method 8260		2.0[b]	See Table III above	
4-Aminobiphenyl	92-67-1	77581	SW-846 Method 8270		2.0[b]	See Table V above	
Atrazine	1912-24-9		SW-846 Method 8270	210 Note 4	2.0[a]	See Table V above	
Benzaldehyde	100-52-7		SW-846 Method 8270	6100 Note 4	2.0[a]	See Table V above	
Benzyl alcohol	100-51-6	75212	SW-846 Method 8270		2.0[a]	See Table V above	
1,1-Biphenyl	92-52-4		SW-846 Method 8270	61.0 Note 4	2.0[a]	See Table V above	
Bromochloromethane (chlorobromomethane)	74-97-5	73085	SW-846 Method 8260		2.0[b]	See Table III above	

Caprolactum	105-60-2		SW-846 Method 8270	31000 Note 4	2.0[a]	See Table V above	
Carbazole	86-74-8	77571	SW-846 Method 8270	24.0 Note 4	2.0[a]	See Table V above	
Carbon Disulfide	75-15-0	78544	SW-846 Method 8260	7800 Note 4	2.0[b]	See Table III above	
p-Chloroaniline (4-Chlorobenzenamine)	106-47-8	73529	SW-846 Method 8270		2.0[b]	See Table V above	
Chlorobenzilate	510-15-6	30381	SW-846 Method 8270 SW-846 Method 8081		2.0[a]	See Table V or VI above	
Chloroprene (2-chloro-1,3-butadine)	126-99-8	81520	SW-846 Method 8260		2.0[a]	See Table III above	
m-Cresol (3-methylphenol)	108-39-4	73133	SW-846 Method 8270		2.0[a]	See Table V above	
o-Cresol (2-methylphenol)	95-48-7	78872	SW-846 Method 8270	310 Note 4	2.0[a]	See Table V above	
p-Cresol (4-methylphenol)	106-44-5	78396	SW-846 Method 8270	31.0 Note 4	2.0[b]	See Table V above	
2,4-D (2,4-dichlorophen-oxyacetic acid)	94-75-7	39731	SW-846 Method 8151		2.0[b]	See Table VI above	
Diallate	2303-16-4	73386	SW-846 Method 8270 SW-846 Method 8081		2.0[a]	See Table V or VI above	
1,2-Dibromo-3-chloropropane (DBCP)	96-12-8	73153	SW-846 Method 8260 SW-846 Method 8270 SW-846 Method 8081	0.08 Note 4	0.08[c]	See Table III, V or VI above	
1,2-Dibromoethane (Ethylene dibromide) (EDB)	106-93-4	79749	SW-846 Method 8260	0.008 Note 4	0.008[c]	See Table III above	
trans-1,4-Dichloro-2-butene	110-57-6	73353	SW-846 Method 8260		2.0[a]	See Table III above	
Dichlorodifluoromethane (CFC 12)	75-71-8	30079	SW-846 Method 8260	490 Note 4	2.0[b]	See Table III above	
Cis-1,2-Dichloroethylene (Cis-1,2-dichloroethene)	156-59-2	77093	SW-846 Method 8260	230 Note 4	2.0[b]	See Table III above	
2,6-Dichlorophenol	87-65-0	73122	SW-846 Method 8270		2.0[a]	See Table V above	

1,3-Dichloropropane (Trimethylene dichloride)	142-28-9	46364	SW-846 Method 8260		2.0[a]	See Table III above	
2,2-Dichloropropane (Isopropylidene chloride)	594-20-7	46366	SW-846 Method 8260		2.0[a]	See Table III above	
1,1-Dichloropropene	563-58-6	46365	SW-846 Method 8260		2.0[a]	See Table III above	
cis-1,3-Dichloropropene	10061-01-5	34704	SW-846 Method 8260	2.0 Note 4	2.0[c]	See Table III above	
0,0-Diethyl 0,2-pyrazinyl phosphorothioate (Thionazin)	297-97-2	73359	SW-846 Method 8270		2.0[a]	See Table V above	
Dimethoate	60-51-5	46303	SW-846 Method 8270		2.0[a]	See Table V above	
p-(Dimethylamino) azobenzene	60-11-7	73116	SW-846 Method 8270		2.0[a]	See Table V above	
7,12-Dimethyl-benz(a)anthracene	57-97-6	73174	SW-846 Method 8270		2.0[a]	See Table V above	
3,3-Dimethylbenzidine	119-93-7	73319	SW-846 Method 8270		2.0[a]	See Table V above	
m-Dinitrobenzene (1,3-dinitrobenzene)	99-65-0	73114	SW-846 Method 8270		2.0[a]	See Table V above	
Dinoseb (DNBP)	88-85-7	38781	SW-846 Method 8270 SW-846 Method 8151		2.0[a]	See Table V or VI above	
Diphenylamine (N-phenylbenzenamine)	122-39-4	30175	SW-846 Method 8270		2.0[a]	See Table V above	
Disulfoton	298-04-4	30001	SW-846 Method 8270		2.0[a]	See Table V above	
Ethyl methacrylate	97-63-2	73126	SW-846 Method 8260		2.0[a]	See Table III above	
Ethyl methanesulfonate	62-50-0	73118	SW-846 Method 8270		2.0[a]	See Table V above	
Famphur	52-85-7	38465	SW-846 Method 8270		2.0[a]	See Table V above	
Hexachloropropene	1888-71-7	73384	SW-846 Method 8270		2.0[a]	See Table V above	

2-Hexanone (Methyl butyl ketone)	591-78-6	75166	SW-846 Method 8260		2.0[b]	See Table III above	
Isobutyl alcohol	78-83-1	73221	SW-846 Method 8260		2.0[a]	See Table III above	
Isodrin	465-73-6	39433	SW-846 Method 8270 SW-846 Method 8081		2.0[a]	See Table V and VI above	
Isosafrole	120-58-1	73321	SW-846 Method 8270		2.0[a]	See Table V above	
Kepone	143-50-0	81857	SW-846 Method 8270		2.0[a]	See Table V above	
Methacrylonitrile	126-98-7	73330	SW-846 Method 8260		2.0[a]	See Table III above	
Methoxychlor	72-43-5	39480	SW-846 Method 8270 SW-846 Method 8081	390 Note 4	2.0[b]	See Table V and VI above	
Methyl Acetate	79-20-9		SW-846 Method 8260	78000 Note 4	2.0[a]	See Table III above	
Methylapyrilene	91-80-5	*PS11	SW-846 Method 8270		2.0[a]	See Table V above	
3-Methylcholanthrene	56-49-5	73156	SW-846 Method 8270		2.0[a]	See Table V above	
Methylcyclohexane	108-87-2		SW-846 Method 8260		2.0[a]	See Table III above	
Methylene Bromide (Dibromomethane)	74-95-3	78756	SW-846 Method 8260		2.0[a]	See Table III above	
Methyl ethyl ketone (MEK) (2-Butanone)	78-93-3	75078	SW-846 Method 8260	3100 Note 4	2.0[b]	See Table III above	
Methyl iodide (Iodomethane)	74-88-4	73121	SW-846 Method 8260		2.0[a]	See Table III above	
Methyl methacrylate	80-62-6	04400	SW-846 Method 8260		2.0[a]	See Table III above	
Methyl methanesulfonate	66-27-3	73190	SW-846 Method 8270		2.0[a]	See Table V above	
2-Methylnaphthalene	91-57-6	78868	SW-846 Method 8270	230 Note 4	2.0[a]	See Table V above	
Methyl parathion (Parathion methyl)	298-00-0	73363	SW-846 Method 8270		2.0[a]	See Table V above	
4-Methyl-2-pentanone; Methyl isobutyl ketone	108-10-1	78133	SW-846 Method 8260		2.0[b]	See Table III above	

Methyl-tert-butyl-ether (MTBE)	1634-04-4	22417	SW-846 Method 8260	110 Note 4	2.0[a]	See Table III above	
1,4-Naphthoquinone (1,4-Naphthalenedione)	130-15-4	73333	SW-846 Method 8270		2.0[a]	See Table V above	
1-Naphthylamine (1-Naphthalenamine)	134-31-7	73143	SW-846 Method 8270		2.0[a]	See Table V above	
2-Naphthylamine (2-Naphthalenamine)	91-59-8	73124	SW-846 Method 8270		2.0[a]	See Table V above	
o-Nitroaniline (2-Nitroaniline) (2-nitrobenzenamine)	88-74-4	78299	SW-846 Method 8270	39.0 Note 4	2.0[a]	See Table V above	
m-Nitroaniline (3-Nitroaniline) (3-nitrobenzenamine)	99-09-2	78869	SW-846 Method 8270		2.0[a]	See Table V above	
p-Nitroaniline (4-Nitroaniline) (4-nitrobenzenamine)	100-01-6	78870	SW-846 Method 8270		2.0[a]	See Table V above	
N-Nitrosodi-n-butylamine	924-16-3	73609	SW-846 Method 8270 SW-846 Method 8260		2.0[a]	See Table III and V above	
N-nitrosodiethylamine	55-18-5	78200	SW-846 Method 8270		2.0[a]	See Table V above	
N-Nitrosomethylethylamine	10595-95-6	73422	SW-846 Method 8270		2.0[a]	See Table V above	
N-Nitrosopiperidine	100-75-4	73619	SW-846 Method 8270		2.0[a]	See Table V above	
N-nitrosopyrrolidine	930-55-2	78206	SW-846 Method 8270		2.0[a]	See Table V above	
5-Nitro-o-toluidine	99-55-8	73127	SW-846 Method 8270		2.0[a]	See Table V above	
Parathion	56-38-2	39540	SW-846 Method 8270		2.0[a]	See Table V above	
Pentachlorobenzene	606-93-5	77793	SW-846 Method 8270		2.0[a]	See Table V above	
Pentachloronitrobenzene	82-68-8	81808	SW-846 Method 8270 SW-846 Method 8081		2.0[a]	See Table V and VI above	
Phenacetin	62-44-2	73117	SW-846 Method 8270		2.0[a]	See Table V above	
p-Phenylenediamine (1,4-Benzenediamine)	106-50-3	73291	SW-846 Method 8270		2.0[a]	See Table V above	

Phorate	298-02-2	46304	SW-846 Method 8270		2.0[a]	See Table V above	
Pronamide	23950-58-5	73031	SW-846 Method 8270		2.0[a]	See Table V above	
Propionitrile (Ethyl cyanide) (Propanenitrile)	107-12-0	73131	SW-846 Method 8260		2.0[a]	See Table III above	
Safrole	94-59-7	73256	SW-846 Method 8270		2.0[a]	See Table V above	
Silvex (2,4,5-TP) [2-(2,4,5-Trichlorophenoxy) propanoic acid]	93-72-1	79732	SW-846 Method 8151		2.0[a]	See Table VI above	
Styrene	100-42-5	81708	SW-846 Method 8260	90.0 Note 4	2.0[b]	See Table III above	
Sulfide, Total (as S)	18496-25-8	00745	SW-846 Method 9030 SW-846 Method 9031 SW-846 Method 9215			7 days Cool to ≤6 degree C Plastic or glass container	
Tertiary butyl alcohol (TBA)	75-65-0	*TBA*	SW-846 Method 8260	1400 Note 4	2.0[a]	See Table III above	
2,4,5-T (2,4,5-Trichloro-phenoxyacetic acid)	93-76-5	39741	SW-846 Method 8151		2.0[b]	See Table VI above	
1,2,4,5-Tetrachlorobenzene	95-94-3	77734	SW-846 Method 8270		2.0[a]	See Table V above	
1,1,1,2-Tetrachloroethane	630-20-6	77562	SW-846 Method 8260		2.0[a]	See Table III above	
2,3,4,6-Tetrachlorophenol	58-90-2	34721	SW-846 Method 8270		2.0[a]	See Table V above	
o-Toluidine	95-53-4	77142	SW-846 Method 8270		2.0[b]	See Table V above	
Trichlorofluoromethane	75-69-4	34488	SW-846 Method 8260	23000 Note 4	2.0[b]	See Table III above	
2,4,5-Trichlorophenol	95-95-4	77687	SW-846 Method 8270	6100 Note 4	2.0[b]	See Table V above	
1,2,3-Trichloropropane	96-18-4	04591	SW-846 Method 8260		2.0[b]	See Table III above	
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	77652	SW-846 Method 8260		2.0[a]	See Table III above	

0,0,0-Triethyl phosphorothioate	126-68-1	73417	SW-846 Method 8270		2.0[a]	See Table V above	
sym-Trinitrobenzene (1,3,5-trinitrobenzene)	99-35-4	73275	SW-846 Method 8270		2.0[a]	See Table V above	
Vinyl acetate	108-05-4	78498	SW-846 Method 8260		2.0[a]	See Table III above	
Xylene (total)	see note 19	81551	SW-846 Method 8260	12000 Note 4	3.0[b]	See Table III above	

Note 1: SM means *Standard Methods for the Examination of Water and Wastewater*, American Public Health Association, 1015 15th Street, NW., Washington, DC 20005.

- Approved versions must be as allowed under 40 CFR Part 136.

SW-846 means *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, EPA Publication SW-846, including all amendments and revisions.

- The most recent method update is recommended; however, any version of the listed method is acceptable provided the laboratory retains New Jersey laboratory certification for the applicable procedure.
- Methods marked with an '*' have been deleted from SW-846 and are scheduled to be phased-out for use by the Department.

EPA/626/R-92/013 means *Environmental Regulations and Technology, Control of Pathogens and Vector Attraction in Sewage Sludge (Including Domestic Septage) Under 40 CFR Part 503*, EPA Publication EPA/625/R-92/013, Revised July 2003. Use the indicated appendix.

EPA Methods: Refer to 40 CFR Part 136 for the published location.

Note 2: **Target Reporting Level** is a performance goal set greater than the lowest, technically feasible detection limit for routine analytical methods and equal to or less than the available regulatory criteria or guidelines. TRLs should be selected such that detection limits reported by the analytical lab are low enough to ensure that the presence of compounds of concern can be ruled in or ruled out at or below the predetermined limit. Typically the lowest value from one of the following was chosen:

[a]Desired regulatory minimum detection levels that are not method specific. (Used when parameter has been found to be typically detected under SQAR monitoring or when there was insufficient data under SQAR to derive a 90th percentile value.)

[b]90th percentile of data submitted under the Sludge Quality Assurance Regulations (N.J.A.C. 7:14C). (Used when parameter has been found to be typically not detected under SQAR monitoring.)

[c]Limit was used.

[d]Highest limits of detection achieved from New Jersey sewage sludge study.

Note 3: Field preservation shall consist of cooling. Chemical preservation in the field is not recommended for sludge samples. Chemical preservation, when applicable, should only be done upon receipt in the certified laboratory performing the analysis.

Note 4: Residential/Non-residential Direct Contact Soil Remediation Standards (N.J.A.C. 7:26D, Appendix 1)

[a] Standard is for Total PCBs.

Note 5: Sludge land application limit; See 40 CFR Part 503 and N.J.A.C. 7:14A-20.7.

Note 6: Lowest sewage sludge incinerator feed sludge quality limitation in Air Pollution Control permits.

Note 7: All samples must be digested using SW-846 Method 3050 or Method 3051 (using an equivalent to 1 gram dry weight) prior to analysis by any of the procedures indicated. See Chapter 6 for additional discussions on limitations on use of available procedures and use of 1 dry gram.

Note 8: Technical Support Document for the Land Application of Sewage Sludge, EPA 822/R-93-001a and 001b, November 1992.

[a]Standard is for Total PCBs.

Note 9: "Environmental Measurements Laboratory Procedures Manual," 28th (1997) or 27th (1990) Editions, Volumes 1 and 2, United States Department of Energy; either edition may be used. In the 27th edition, the Gamma Spectrometry Method Ga-01-R is listed as Section 4.5.2.3. This method can be used for the following parameters: Actinium 228; Bismuth 214; Cesium 137; Lead 210; Lead 212; Lead 214; Potassium 40; Radium 226; Radium 224; Thallium 208; Thorium 232; Thorium 234; Uranium 234; Uranium 235; Uranium 238.

Note 10: The samples shall be prepared (e.g., weighed, dried, ground, blended, sealed, held for 21 days and counted pursuant to the procedures recommended by the ASTM (e.g., ASTM Standard C999-83, C998-83, D3649-78 and D4220-83), the USEPA (SW-846), the Environmental Measurements Laboratory (HASL-300), or technically equivalent methods.

Note 11: USEPA. July 2006. Method 1682: *Salmonella* in Sewage Sludge by Modified Semisolid Rappaport-Vassiliadis (MSRV) Medium. U.S. Environmental Protection Agency, Office of Water, Washington DC EPA-821-R-06-014.

Note 12: Recommended for enumeration of target organism in sewage sludge.

Note 13: 40 CFR Part 136, Table II. For fecal coliform samples for sewage sludge, the holding time is extended to 24 hours for the following sample types using either EPA Method 1680 or 1681: Class A composted, Class B anaerobically digested, or Class B aerobically digested.

Note 14: PA is any plastic that is made of a sterlizable material (polypropylene or other autoclavable plastic).

Note 15: USEPA. July 2010. Method 1680: Fecal Coliforms in Sewage Sludge by Multiple-Tube Fermentation Using Lauryl-Tryptose Broth (LTB) and EC Medium. U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA-821-R-10-003.

Note 16: USEPA. July 2006. Method 1681: Fecal Coliforms in Sewage Sludge by Multiple-Tube Fermentation using A-1 Medium. U.S. Environmental Protection Agency, Office of Water, Washington DC EPA-821-R-06-013.

Note 17: Page 70, Table 9-2. *Environmental Regulations and Technology, Control of Pathogens and Vector Attraction in Sewage Sludge Under 40 CFR Part 503*, EPA Publication EPA/625/R-92/013, Revised July 2003.

Note 18: EPA Publication SW-846, Method 9056 recommends a 48 hour holding time for Nitrate-Nitrogen. However, a longer holding time is recommended based on the discussion presented in Chapter 6 of this Guidance Document.

Note 19: Xylene (total): This entry includes o-xylene (CAS RN 96-47-6), m-xylene (CAS RN 108-38-3), p-xylene (CAS RN 106-42-3), and unspecified xylenes (dimethylbenzenes) (CAS RN 1330-20-7).

APPENDIX B

Dioxin and Dioxin-like Compounds

TABLE 1

(Analysis of 17 Polychlorinated DibenzoDioxins and DibenzoFurans)

(Using USEPA Method 1613B)

#	CASRN	TEF	Compound Name
1	1746-01-6	1.0	2,3,7,8-Tetrachlorodibenzo-p-dioxin
2	40321-76-4	1.0	1,2,3,7,8-Pentachlorodibenzo-p-dioxin
3	39227-28-6	0.1	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin
4	57653-85-7	0.1	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin
5	19408-74-3	0.1	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin
6	35822-46-9	0.01	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin
7	3268-87-9	0.0003	1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin
8	51207-31-9	0.1	2,3,7,8-Tetrachlorodibenzofuran
9	57117-41-6	0.03	1,2,3,7,8-Pentachlorodibenzofuran
10	57117-31-4	0.3	2,3,4,7,8-Pentachlorodibenzofuran
11	70648-26-9	0.1	1,2,3,4,7,8-Hexachlorodibenzofuran
12	57117-44-9	0.1	1,2,3,6,7,8-Hexachlorodibenzofuran
13	72918-21-9	0.1	1,2,3,7,8,9-Hexachlorodibenzofuran
14	60851-34-5	0.1	2,3,4,6,7,8-Hexachlorodibenzofuran
15	67562-39-4	0.01	1,2,3,4,6,7,8-Heptachlorodibenzofuran
16	55673-89-7	0.01	1,2,3,4,7,8,9-Heptachlorodibenzofuran
17	39001-02-0	0.0003	1,2,3,4,6,7,8,9-Octachlorodibenzofuran

TABLE 2

(Analysis of 12 PCB Congeners)

(Using USEPA Method 1668A)

#	*CASRN*	TEF	*PCB Name*	*IUPAC #*
1	32598-13-3	0.0001	3,3’,4,4’-Tetrachlorobiphenyl	77
2	70362-50-4	0.0003	3,4,4’,5-Tetrachlorobiphenyl	81
3	32598-14-4	0.00003	2,3,3’4,4’-Pentachlorobiphenyl	105
4	57465-28-8	0.1	3,3’,4,4’,5-Pentachlorobiphenyl	126
5	31508-00-6	0.00003	2,3’4,4’,5-Pentachlorobiphenyl	118
6	65510-44-3	0.00003	2’3,4,4’,5-Pentachlorobiphenyl	123
7	74472-37-0	0.00003	2,3,4,4’,5-Pentachlorobiphenyl	114
8	32774-16-6	0.03	3,3’,4,4’,5,5’-Hexachlorobiphenyl	169
9	38380-08-4	0.00003	2,3,3’4,4’,5-Hexachlorobiphenyl	156
10	69782-90-7	0.00003	2,3,3’4,4’,5’-Hexachlorobiphenyl	157
11	52663-72-6	0.00003	2,3’,4,4’,5,5’-Hexachlorobiphenyl	167
12	39635-31-9	0.00003	2,3,3’4,4’,5,5’-Heptachlorobiphenyl	189

APPENDIX C

Selecting a Certified Laboratory

The following information is provided to assist in selecting a laboratory to conduct your sludge sample analyses. This list includes only some of the potential questions that could be asked. Site-specific concerns may alter the direction of your questions. *Source: The Wastewater Treatment Plant Operators Guide to Biosolids Sampling Plans, Appendix E.* New England Interstate Water Pollution Control Commission, 116 John Street, Lowell, Ma 01852. September 2006. A copy of the Guide is available at: *http://www.neiwpcc.org/biosamplingguide.asp*

General Feasibility

- Is the lab certified by the State of New Jersey, NJDEP, Office of Quality Assurance for the methods and matrices requested?
- Does the lab routinely perform sludge analyses using the required/requested analyses? Are individuals qualified and do they have written qualifications available?
- Is the lab's turn-around time compatible with your schedule?
- Will the geographical location of the lab cause additional expense (telephone and shipping) and potential difficulty in communication?

Concerns Prior to Sample Collection and Shipment

- Will the lab provide coolers and sample containers?
- What type of sample chain-of-custody is commonly used and will the lab provide chain-of-custody forms prior to shipment?
- What form of shipment is commonly used (Federal Express, UPS, other)? Will the lab pay the shipment cost?
- On what days will someone be available to receive sample shipments (Saturday)?
- What type of sample container should be used and does the lab have any specific packaging requirements?

Costs

- How will I be billed (invoice, pre-pay, other) – NEVER PRE-PAY FOR ANALYSES.
- Are sample containers provided for free or are there additional costs involved?
- What are the sample preparation costs and when and how are they incurred (per sample, per analysis, other)?

- Are there any additional costs involved, which I may not be aware of at this time?
- Can a written estimate be provided and what factors might cause the actual price to differ from the estimate?
- Will QA/QC of my samples involve additional cost?

Data Processing

After the analyses have been conducted, the lab will provide you with a data package summarizing the analyses. The data package can differ greatly from lab to lab, so the following questions should be asked prior to sample shipment. If any of the information below is not included in the data package, ask the lab to provide it.

- What type of report will I receive? Will narrative descriptions be provided for help in evaluating the data package?
- Will the data be presented on a dry weight basis? IF NOT, REQUIRE IT.
- Will I receive a QA/QC report along with the data package?
- If data qualifiers are present, will a key be provided?
- Will the detection limits for each analysis be provided?
- Will sample collection times be reported?
- Will the dates and times of all analyses be reported?
- Will the analytical methods used be included?

APPENDIX D

Sampling Plan Template

Each domestic or industrial treatment works shall develop and maintain on file a sludge sampling plan that details its sampling and analytical procedures (SQAR at N.J.A.C. 7:14C-1.6).

The following information is provided to assist you in developing a sludge sampling plan. This list includes only some of the potential questions that could be asked. Site-specific concerns may alter the direction of your questions. The following document was used to assist in development of this Template: *The Wastewater Treatment Plant Operators Guide to Biosolids Sampling Plans, Appendix E*. New England Interstate Water Pollution Control Commission, 116 John Street, Lowell, Ma 01852. September 2006. A copy of the Guide is available at: *http://www.neiwpcc.org/biosamplingguide.asp*

1. General Facility Information

   Facility Name:

   Contact: Title:

   Phone:

   Street Address:

   City: State: Zip:

2. Describe each treatment unit where solids are generated, the wasting schedule for each location where solids are generated and each subsequent intrafacility location where solids are transferred for processing or treatment (include in the description the point where any chemicals are added and the type of chemical that is added):

3. Describe the intended sampling location(s) and the rationale for choosing such location(s) (Where a treatment works generates different types of sludge that are removed separately for use or disposal, or where a treatment works accepts customer sludge or septage, separate sampling points for each different type of sludge may need to be established):

4. Describe the sampling equipment to be used (sampling device, container type and size, and container cover):

5. Describe the procedure to be used for cleaning/decontamination of sample containers and sampling equipment (see Chapter 4):

6. Describe in detail the procedure to be used for collecting the sample(s) to ensure the sample obtained for analysis is representative of the sludge removed for use or disposal, including a schedule for days and times of sample collection, the procedures to be used to obtain a representative sample from the chosen sampling point, and the procedures to be used to mix composite samples (See SOP in Appendix E):

7. Describe the sampling method(s) (that is, Grab v. Composite), the number of samples to be taken per sampling event and the interval between grabs (include sample size.) (Note, different parameters or groups of parameters may require different sampling methods and/or locations.):

8. Provide the name of the person who will take the sample(s) and his/her qualifications:

9. Provide the frequency of analysis and the analytical methods requested for each required parameter. Note sample holding times and Target Reporting Level for use with your certified laboratory (see *New Jersey Sludge Sampling and Analytical Guidance Document*, Appendix A):

PARAMETER	FREQUENCY OF ANALYSIS	ANALYTICAL METHOD	HOLDING TIME	TARGET REPORTING LEVEL

10. Describe the post-collection sample handling procedures employed to maintain sample integrity. This description should explain how the samples will be preserved and transported,

what the appropriate hold-time is for each analysis, and whether a chain-of-custody is required (See SOP in Appendix E):

11. Describe sample documentation procedures, specifically, describe those elements to be included in a field logbook (see Appendix F):

12. In addition to Item number 9 above, describe other elements of the sampling event that need to be conveyed to the certified laboratory (for example, what chemicals have been added during sludge processing, treatment process conditions or deviations):

13. Provide a description of record-keeping procedures. The description should explain what information will be retained and for how long, how the information will be stored, and what records are required to be reported:

APPENDIX E

EXAMPLE
Sludge Sampling Standard Operating Procedures

The following example is provided to assist in developing sludge collection and handling procedures. This list includes only some of the potential elements to include. Site-specific concerns may alter the direction of individual operating procedures. The following document was used to assist in development of the SOP: *The Wastewater Treatment Plant Operators Guide to Biosolids Sampling Plans, Appendix E*. New England Interstate Water Pollution Control Commission, 116 John Street, Lowell, Ma 01852. September 2006. A copy of the Guide is available at: *http://www.neiwpcc.org/biosamplingguide.asp*

1. A week to several days prior to the proposed sampling, confirm or schedule sludge processing (dewatering and treatment) to ensure that sludge in the appropriate form (liquid versus dewatered, untreated cake versus treated biosolids) is available for sampling at the proposed date, time, and sampling point.
2. A week to several days prior to the proposed sampling date, schedule/confirm that contract lab performing the analyses is ready and willing to accept samples on the proposed sampling date.
3. At least one day before collecting samples, assemble the equipment necessary to accomplish the proposed sampling. Ensure that all equipment is clean and in good working order.
4. On the day of sampling, obtain ice for sample storage and transportation and place in sample coolers.
5. After arrival at the sampling location/sampling point (as determined in the sampling plan), evaluate the operation of the sludge handling train (dewatering, biosolids treatment, etc.). Any observable deviations from normal operation should be noted in Field Logbook prior to collecting samples.
6. Put on nitrile gloves and any other required/desired personal safety equipment.
7. To collect a composite sample for metals, TKN, NH3, and NO3 analyses, take the first of 8 grab samples from the belt filter press as biosolids fall into the roll-off container. All grab samples should be collected using a 500 mL Teflon beaker and a stainless steel trowel, and should be approximately 200 mL in volume. After collecting each grab sample, place the sample in the stainless steel bucket and record the time of collection. Wait one hour and collect the next grab sample. Repeat the process until all eight grab samples are collected. Between collection of grab samples, the previously collected

material should be kept cool (at or around 4 degrees Celsius). Ensure that any required or planned field duplicates or blanks are also collected.

8. After the first grab has been collected, it should be placed in a stainless steel bucket. A subsample is placed in a 125 mL glass container and filled as full as practical in order to minimize headspace. This sample should be placed on ice and cooled until analyzed according to EPA Method 8260 for volatile organic compounds (VOC).
9. After the first grab sample, another grab sample should be collected every 30 minutes and placed in the stainless steel bucket until all 8 grab samples have been collected. Again, the grab samples should be of approximately equal size (weight or volume). During the time between samples, the stainless steel bucket should be covered and placed on ice or refrigerated. (This is necessary whenever the interval between grab samples is longer than five minutes.) The time of collection of the last grab sample should be recorded.
10. Once the last grab sample has been collected, thoroughly mix all material accumulated in the stainless steel bucket using a stainless steel trowel. The goal of the mixing process is to produce a homogeneous sample. After the material is completely mixed, record the current time as the composite sample collection time.
11. After mixing, label all sample containers with the following minimum information:
    a) Sample Identification (ID) Number
    b) Facility name
    c) Date and time of collection
    d) Sample location
    e) Type of sample (for example, 5-grab composite)
    f) Person collecting sample
    g) Required test(s)
12. After labeling, fill each individual sample container with portions of the homogenized sample within the stainless steel bucket.
13. After each sample container is filled, seal it with a signed custody seal and place on ice in a cooler for transportation to the laboratory.
14. Prior to delivering the samples to the lab, complete a chain-of-custody sheet to document proper sample handling.
15. After sample delivery, clean all equipment according to established procedures and store in a clean, dry area.

APPENDIX F

Logbook Template

The following information is provided to assist in developing a field logbook to document sludge sampling activities. This list includes only some of the potential information that could be included. Sampling situations vary widely. Therefore, no general rule can be given as to the extent of information that must be entered in the logbook. A good rule, however, is to record sufficient information so that anyone can reconstruct the sampling without reliance on the collector's memory.

1. General Facility Information

Facility Name: _____

Street Address: _____

City: _____ State: _____ Zip: _____

2. Sampling Personnel:

Individual(s) that performed sampling: _____

Title: _____ Phone: _____

3. Sample Documentation:

Date(s) of sampling: _____

Time of sampling or measurement: (Begin):_____ (End): _____

Exact location of sampling: _____

Type of sample collected (composite or grab): _____

Number of grab samples and volume taken: _____

Interval between grab samples and their relative weighting: _____

Sample Identification Numbers: _____

4. Observations:

The weather conditions at the time of sampling (including any unusual hot/cold or wet/dry spells): _____

Deviations from established protocols: _____

Other observations which could potentially impact the laboratory analytical results, including any operational changes since the last sampling event (for example, changes in polymer use):
_____

_____

5. Sampling Procedures:

Sampling equipment and a brief description of sampling procedures: _____

_____

_____

6. **Sample transportation method** (e.g. name of laboratory, UPS, Federal Express):
_____

7. **Signatures of personnel responsible for observations**:

Name (Print): _____

Name (Sign): _____